IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
J. Bednorz et al. : Date: December 15, 1998

Serial No. 08/303,561 : Group Art Unit: 1105

Filed: September 9, 1994 : Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, David B. Mitzi, being duly sworn, do hereby depose and state:

That I received a B. S. E. degree in Electrical Engineering/Engineering Physics (1985) from Princeton University and a PhD. degree, in Applied Physics (1990) from Stanford University, California.

That I have worked as a research staff member in Solid State Chemistry at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, NY from 1990 to the present.

That I have worked in the fabrication of and characterization of high temperature superconductor and related materials from 1990 to the present.

That I have reviewed the above-identified patent application and that I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and

Muller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Muller behave in a similar manner, conduct current in a similar manner and have similar magnetic properties.

That once a person of skill in the art knows of a specific transition metal oxide composition which is superconducting above 26°K, such a person of skill in the art, using the techniques described in the above-identified patent application, which includes all knows principles of ceramic fabrication known at the time the application was filed, can make the transition metal oxide compositions encomposed by the claims in the above identified application, without undue experimentation or without requiring ingenuity beyond that expected of a person of skill in the art. This is why the work of Bednorz and Muller was reproduced so quickly after their discovery and why so much additional work was done in this field within a short period of their discovery.

The general principles of ceramic science referred to by Bednorz and Mueller in their patent application can be found in many books and articles published before their discovery. An exemplary list of books describing the general principles of ceramic fabrication are:

1) Introduction to Ceramics, Kingery et al., Second Edition, John Wiley & Sons, 1976, in particular pages 5-20, 269-319, 381-447 and 448-513, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

2) Polar Dielectrics and Their Applications, Burfoot et al., University of California Press, 1979, in particular pages 13-33, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-186, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

An exemplary list of articles applying their general principles of ceramic fabrication to the types of materials described in applicants' specification are (these references are cited on applicant's 1449 form submitted August 5, 1987 and in PTO Form 892 in Paper # 20, Examiner's action dated August 8, 1990):

1) Oxygen Defect K_2NiF_4 - Type Oxides: The Compounds La_{2-x} $Sr_xCuO_{4-x/2+\delta}$, Nguyen et al., Journal of Solid State Chemistry 39, 120-127 (1981).

2) The Oxygen Defect Perovskite $BaLa_4$ $Cu_5O_{13+\delta}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}$ $B_{0.5})$ O_3, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: David B. Mitzi

David B. Mitzi

Sworn to before me this 15th day of December, 1998

Notary Public

DANIEL P. MORRIS
NOTARY PUBLIC, State of New York
No. 4888676
Qualified in Westchester County
Commission Expires March 16, 19 99

YO987-074BY

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
J. Bednorz et al. : Date: December 15, 1998

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AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, Timothy Dinger, being duly sworn, do hereby depose and state:

That I received a B. S. degree in Ceramic Engineering (1981) from New York State College of Ceramics, Alfred University, an M. S. degree (1983) and a PhD. degree (1986), both in Material Science from the University of California at Berkley.

That I have worked as a research staff member in Material Science at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, NY from 1986 to the present.

That I have worked in the fabrication of and characterization of high temperature superconductor materials from 1987 to 1991.

That I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Muller, which is generally recognized as the first discovery of

superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Muller behave in a similar way, conduct current in a similar manner and have similar magnetic properties.

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1) Introduction to Ceramics, Kingery et al., Second Edition, John Wiley & Sons, 1976, in particular pages 5-20, 269-319, 381-447 and 448-513, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998

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1) Oxygen Defect K_2NiF_4 - Type Oxides: The Compounds La_{2-x} Sr_x $CuO_{4-x/2+\bullet}$, Nguyen et al., Journal of Solid State Chemistry 39, 120-127 (1981).

2) The Oxygen Defect Perovskite $BaLa_4$ Cu_5-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}$ $B_{0.5})$ 0_3, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Timothy R. Dinger
Timothy Dinger

Sworn to before me this 16th day of December, 1998

Sandra M. Emma
Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
J. Bednorz et al. : Date: December 15, 1998

Serial No. 08/303,561 : Group Art Unit: 1105

Filed: September 9, 1994 : Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

AFFIDAVIT UNDER 37 C.F.R. 1.132

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Sir:

I, Chang C. Tsuei, being duly sworn, do hereby depose and state:

That I received a B. S. degree in Mechanical Engineering from National Taiwan University (1960) and M. S. and PhD. degrees, in Material Science (1963, 1966) respectively from California Institute of Technology.

That I have worked as a research staff member and manager in the physics of superconducting, amorphous and structured materials at the Thomas Watson Research Center of the International Business Machines Corporation in Yorktown Heights, New York from 1973 to the present. (See attached Exhibit A for other professional employment history.)

That I have worked in the fabrication of and characterization of high temperature superconductor and related materials from 1973 to the present.

That I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Muller, which is generally recognized as the first discovery of

superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Muller behave in a similar manner, conduct current in a similar manner and have similar magnetic properties.

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2) Polar Dielectrics and Their Applications, Burfoot et al., University of California Press, 1979, in particular pages 13-33, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-186, a copy of which is with the Affidavit of Thomas Shaw submitted December 15, 1998.

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2) The Oxygen Defect Perovskite $BaLa_4$ Cu_5-$0_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}$ $B_{0.5})$ 0_3,V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Chang C. Tsuei

Chang C. Tsuei

Sworn to before me this 16th day of December, 1998

Sandra M. Emma

Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

YO987-074BY

CHANG C. TSUEI

Education

California Institute of Technology, M.S. (1963), Ph.D. (1966)

National Taiwan University, B.S. (1960)

Professional Employment

1993 - present - Research Staff Member

1983 - 1993 - Manager, Physics of Structured Materials

1979 - 1983 - Manager, Physics of Amorphous Materials

1974 - 1975 - Acting Manager, Superconductivity

1973 - 1979 - Research Staff Member

Harvard University: 1980 (Summer)

Visiting Scholar in Applied Physics

Stanford University: 1982 (Sept.) - 1983 (April)

Visiting Scholar in Applied Physics

California Institute of Technology

1972 - 1973 - Senior Research Associate in Applied Physics

1969 - 1972 - Senior Research Fellow in Materials Science

1966 - 1969 - Research Fellow in Materials Science

Exhibit A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: J. Bednorz et al.

Date: December 15, 1998

Serial No. 08/303,561

Group Art Unit: 1105

Filed: September 9, 1994

Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

The Commissioner of Patents and Trademarks
Washington, D.C. 20231

AFFIDAVIT UNDER 37 CFR 1.132

Sir:

I, Thomas M. Shaw, being duly sworn, do hereby depose and state:

I received a B.S. degree in Metallurgy from the University of LIverpool, Liverpool, England and a M.S. and PhD. degree in Materials Science (1981) from the University of California, Berkeley.

I have worked as a postdoctoral researcher in the Material Science Department of Cornell University from 1981-1982. I worked at Rockwell International Science Center in Thousand Oaks, California from 1982-1984 as a ceramic scientist. I have worked as a research staff member in Ceramics Science at the Thomas J. Watson Research

Center of the International Business Machines Corporation in Yorktown Heights, N.Y. from 1984 to the present.

I have worked in the fabrication of and characterization of ceramic materials of various types, including superconductors and related materials from 1984 to the present.

Attached is a resume of my publications. I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Mueller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

That all the high temperature superconductors which have been developed based on the work of Bednorz and Mueller behave in a similar manner, conduct current in a similar manner and have similar magnetic properties.

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3) Ceramic Processing Before Firing, Onoda et al., John Wiley & Sons, 1978, the entire book, a copy of which is attached herewith.

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2) The Oxygen Defect Perovskite $BaLa_4Cu_5O_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5} B_{0.5}) O_3$. V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

By: Thomas M. Shaw
Thomas M. Shaw

Sworn to before me this 14th day of December, 1998.

Sandra M. Emma
Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: J. Bednorz et al.

Date: December 18, 1998

Serial No. 08/303,561

Group Art Unit: 1105

Filed: September 9, 1994

Examiner: M. Kopec

For: NEW SUPERCONDUCTIVE COMPOUNDS HAVING HIGH TRANSITION TEMPERATURE, AND METHODS FOR THEIR USE AND PREPARATION

The Commissioner of Patents and Trademarks
Washington, D.C. 20231

AFFIDAVIT UNDER 37 CFR 1.132

Sir:

I, Peter R. Duncombe, being duly sworn, do hereby depose and state:

I received a B.A. degree in Chemistry from the State University of New York at New Paltz, New Paltz, N.Y. and a M.S. degree in Chemical Engineering (1983) from the State University of New York at Buffalo, Buffalo, N.Y.

I have worked as a graduate research assistant in the Chemical Engineering Department of SUNY at Buffalo from 1980-1983. I have worked as a chemical engineer in Ceramics Science at the Thomas J. Watson Research Center of the International Business Machines Corporation in Yorktown Heights, N.Y. from 1984 to the present.

I have worked in the fabrication of and characterization of ceramic materials of various types, including superconductors and related materials from 1984 to the present.

Attached is a resume of my publications (Attachment A).

I have reviewed the above-identified patent application and acknowledge that it represents the work of Bednorz and Mueller, which is generally recognized as the first discovery of superconductivity above 26°K and that subsequent developments in this field have been based on this work.

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4) Structure, Properties and Preparation of Perovskite-Type Compounds, F.S. Glasso, Pergamon Press, 1969, in particular pages 159-181, a copy of which is attached herewith.

An exemplary list of articles applying their general principles of ceramic fabrication to the types of materials described in applicants' specification are (these references are cited on applicant's 1449 form submitted August 5, 1987 and in PTO Form 892 in Paper # 20, Examiner's action dated August 8, 1990):

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2) The Oxygen Defect Perovskite $BaLa_4Cu_5O_{13.4}$, A Metallic Conductor , C. Michel et al., Mat. Res. Bull., Vol. 20, pp. 667-671, 1985.

3) Oxygen intercalation in mixed valence copper oxides related to the perovskite, C. Michel et al., Revue de Chemie minerale, p. 407, 1984.

4) Thermal Behaviour of Compositions in the Systems x $BaTiO_3$ + (1-x) $Ba(Ln_{0.5}\ B_{0.5})\ O_3$, V.S. Chincholkar et al. Therm. Anal. 6th, Vol. 2., p. 251-6, 1980.

I have recorded research notes relating to superconductor oxide (perovskite) compounds in technical notebook IV with entries from November 12, 1987 to June 14, 1988 and in technical notebook V with entries continuing from June 7, 1988 to May 2, 1989. Complete copies of each of these notebooks are attached - Attachment B - Book IV and Attachment C - Book V. Below is a listing of some of the compounds I prepared and recorded in these notebooks according to the teaching as described in the Bednorz and Mueller patent application using the general principles of ceramic science as described in the books and articles listed above.

In Book IV, $Y_1Ba_2Cu_3O_x$ batch C1 pellet pressing, sintering notes and powder processing specifications start on page 2 and continue intermittently to pg. 40 (pg. 13 has superconductive susceptibility curves for pellet 9). Batch C2 $Y_1Ba_2Cu_3O_3$ detailed from pages 14 to 47.

In Book V green phase (Y_2BaCuO_x) microstructural photomicrographs are logged on pages 15-17 with notes continuing to pg. 19. The perovskite superconductor BiSrCaCu oxide ($Bi_{2.15}Sr_{1.68}Ca_{1.7}Cu_2O_{8+\delta}$) and related perovskites $Ca_{(2-x)}Sr_xCuOx$ and $Bi_2Sr_2CuO_x$ synthesis notations start and continue through pg. 61 with microstructural photomicrographs.

A series of $Y_1Ba_2Cu_3O_x$ stoichiometric perturbations to study compositional effects on 2nd phase or grain boundary phases and their effect on conductivity (resistivity), sintering behavior etc., continue until the end of the book notes on the page dated May 2, 1989 (page not numbered). These are typical perovskite synthetic procedures, microstructural photomicrographs, powder processing methods, characteristic susceptibility curve(s), sintering behavior and the like. Additional notes may be available in later notebooks.

The undersigned affiant swears further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or patent issuing thereon.

By: Peter R. Duncombe
Peter R. Duncombe

Sworn to before me this 18th day of December, 1998.

Sandra M. Emma
Notary Public

SANDRA M. EMMA
Notary Public, State of New York
No. 01PO4935290
Qualified in Westchester County
Commission Expires July 5, 2000

ATTACHMENT A

RESUME 1998

1. Compensation doping of Ba0.7Sr0.3TiO3 thin films
   Copel, M Baniecki, JD Duncombe, PR Kotecki, D
   Laibowitz, R Neumayer, DA Shaw, TM
   APPLIED PHYSICS LETTERS V73 N13 SEP 28 1998 P1832-1834

2. Method for Forming Noble Metal Oxides and Structures Formed Thereof. June 1998.
   Duncombe, P. R. Hummel, J. P. Laibowitz, R. B.
   Neumayer, D. A. Saenger, K. L. Schrott, A. G.
   RC 98A 41575

3. Growth of Bismuth Titanate Films By Chemical Vapor Deposition and Chemical Solution Deposition. March 1998. RC-21124
   Neumayer, D. A. Duncombe, P. R. Laibowitz, R. B.
   Shaw, T. Purtell, R. Grill, A.

4. Dielectric relaxation of Ba0.7Sr0.3TiO3 thin films from 1 mHz to 20 GHz Baniecki, JD
   Laibowitz, RB Shaw, TM Duncombe, PR
   Neumayer, DA Kotecki, DE Shen, H Ma, QY
   APPLIED PHYSICS LETTERS V72 N4 JAN 26 1998 P498-500

5. Contrasting magnetic and structural properties of two La manganites with the same doping levels
   McGuire, T.R. Duncombe, P.R. Gong, G.Q. Gupta, A. Li, X.W. Pickart, S.J. Crow, M.L.
   J. Appl. Phys. (USA) Vol.83, No.11 1 June 1998 P7076-8

6. Effects of Annealing Conditions on Charge Loss Mechanisms in MOCVD (Ba0.7,Sr0.3)TiO3 Thin Film Capacitors.
   Baniecki, J.D., Laibowitz, RB Shaw, TM Duncombe, PR Saenger, KL Cabral C
   Kotecki, DE , Shen, H , Lian, J., Ma, QY

7. Low Operating Voltage and High Mobility Field Effect Transistors Comproising Pentacene and Relatively High Dielectric Constant Insulators RC21233(94806) 7/17/98
   Dimitrakopoulos, CD Purushothaman S , Kymissis J. Callegari A. , Neumayer DA,
   Duncombe PR, Laibowitz RB, Shaw JM

8. Maximum Magnetorsistance in Granular Manganite/Insulator System close to Percolation Threshold PACS 10/06/98
   DK Petrov, L Krusin-Elbaum, JZ Sun, C Feild, & PR Duncombe

9. Magnetorsistance and Hall Effect of Chromium Dioxide Epitaxial Thin Films
   X.W. Li, A. Gupta, T.R. McGuire, P.R. Duncombe, Gang Xiao

10. Progress Report on High-k dielectric material: amorphous BST from solgel (09/98)
    P. Andry, D. Neumayer, P. Duncombe, C. Dimitrakopoulos, F. Libsch, A. Grill, R. Wisnieff

RC21352(96175)2 Dec 1998

INCOMPLETE

Personal Inventor History

Name:Duncombe, P.R. Serial:155139 Loc:RES YORKTOWN
Patent Pts:36 TDB Pts:1 Total Pts:37 Plateau Lvl:3
Plateau Date:10/24/98 File Update:11/02/98
Awards Due:None

Title: NOVEL METAL ALKOXYALKOXIDECARBOXYLATES AND USE TO FORM FILMS
06/17/98 Opened as Discl YO8980231 Status:Filed
06/22/98 Discl Review Action:File
(1) 09/04/98 Filed as Docket YO998254 in US Rating: 2 Pts:3
Co-inventors: Neumayer, D.A.

Title: SELECTIVE GROWTH OF FERROMAGNETIC FILMS FOR MAGNETIC MEMORY, STORAGE-BASED DEVICES, AND OTHER DEVICES
06/17/98 Opened as Discl YO8980225 Status:Filed
06/29/98 Discl Review Action:File
(4) 10/15/98 Filed as Docket YO998268 in US Rating: 2 Pts:3
Co-inventors: Guha, S. Gupta, A. Bojarczuk, N.A. Karasinski, J.M.

Title: BEOL DECOUPLING CAPACITOR MATERIALS
01/28/98 Opened as Discl YO8980024 in US Status:Opened
06/24/98 Discl Review Action:File
Co-inventors: Rosenberg, R. Ning, T.H. Shaw, T.M. Edelstein, D.C. Neumayer, D.A. Laibowitz, R.B.

(3) "FABRICATION OF STRONTIUM BISMUTH TANTALATE/BISMUTH TITANATE MULTILAYER FERROELECTRIC"
Title: FERROELECTRIC THIN FILM STRUCTURES
10/01/97 Opened as Discl YO8970512 in US Status:Opened
09/16/98 Discl Review Action:File
Co-inventors: Shaw, T.M. Neumayer, D.A. Laibowitz, R.B.

(2) 10/30/98 SENT TO COUNSEL (L. Schiros)

Title: CAPACITORS WITH AMORPHOUS DIELECTRICS AND IMPROVED DIELECTRIC PROPERTIES MADE USING SILICON SURFACES AS ELECTRODES
06/06/97 Opened as Discl YO8970261 in US Status:Opened
Co-inventors: Shaw, T.M. Neumayer, D.A. Laibowitz, R.B.

Title: FABRICATION OF THIN FILM FIELD EFFECT TRANSISTOR COMPRISING AN ORGANIC SEMICONDUCTOR AND CHEMICAL SOLUTION DEPOSITED METAL OXIDE
03/25/97 Opened as Discl YO8970113 Status:Filed
03/25/97 Discl Review Action:File
03/25/97 Filed as Docket YO997083 in US Rating: 2 Pts:3
(6) 03/24/98 Filed as Docket YO997083 in JA Rating: 2
03/16/98 Filed as Docket YO997083 in TA Rating: 2
03/12/98 Filed as Docket YO997083 in KO Rating: 2
04/24/98 Last Office Action
Co-inventors: Purushothaman, S. Dimitrakopoulos, C.D. Furman, B.K. Neumayer, D.A. Laibowitz, R.B.

Title: NOVEL ALKOXYALKOXIDES AND USE TO FORM FILMS
10/30/96 Opened as Discl YO8960411 Status:Filed
03/10/97 Discl Review Action:File
(5) 01/30/98 Filed as Docket YO997069 in US Rating: 2 Pts:3
Co-inventors: Neumayer, D.A.

Title: THIN-FILM FIELD-EFFECT TRANSISTOR WITH ORGANIC SEMICONDUCTOR REQUIRING LOW OPERATING VOLTAGES
09/11/96 Opened as Discl YO8960358 Status:Filed
03/04/97 Discl Review Action:File
(7) **03/25/97** Filed as Docket YO997057 in US Rating: 2 Pts:3
03/12/98 Filed as Docket YO997057 in KO. Rating: 2
04/10/98 Last Office Action
Co-inventors: Purushothaman, S. Dimitrakopoulos, C.D. Furman, B.K. Neumayer, D.A. Laibowitz, R.B.

X **Title:** HIGH DIELECTRIC CONSTANT, BARIUM LANTHANUM TITANATE THIN FILM CAPACITORS FOR RANDOM ACCESS
06/20/96 Opened as Discl YO8960255 in US Status:Opened
Co-inventors: Gupta, A. Shaw, T.M. Laibowitz, R.B.

Title: METHOD FOR FORMING NOBLE METAL OXIDES AND STRUCTURES FORMED THEREOF
10/30/95 Opened as Discl YO8950450 Status:Filed
11/12/96 Discl Review Action:File
11/05/97 Filed as Docket YO996239 in US Rating: 2 **Pts:**3
(8) **10/20/98** Filed as Docket YO996239 in JA Rating: 2
07/30/98 Filed as Docket YO996239 in TA Rating: 2
Co-inventors: Schrott, A.G. Saenger, K.L. Hummel, J.P. Neumayer, D.A. Laibowitz, R.B.

Title: PEROXIDE ETCHANT PROCESS FOR PEROVSKITE-TYPE OXIDES
10/23/95 Opened as Discl YO8950434 Status:Filed
08/08/97 Discl Review Action:File
(9) **04/08/98** Filed as Docket YO997256 in US Rating: 2 Pts:3
Co-inventors: Rosenberg, R. Cooper, E.I. Laibowitz, R.B.

Title: RF TRANSPONDER FOR METALLIC SURFACES
08/02/95 Opened as Discl YO8950329 in US Status:Opened
Co-inventors: Afzali-ardakani, A. Feild, C.A. Duan, D.W. Brady, M.J. Moskowitz, P.A.

Title: METHOD FOR CLEANING THE SURFACE OF A DIELETRIC
09/06/95 Opened as Discl FI8950292 Status:Filed
09/06/95 Sent to Evaluator
02/05/96 Evaluated Action:Search
04/19/96 Discl Review Action:File
12/06/96 Filed as Docket FI996047 in US Rating: 2 Pts:3
11/29/97 Filed as Docket FI996047 in KO Rating: 2
05/26/97 Filed as Docket FI996047 in TA Rating: 2
06/11/98 Last Office Action
Co-inventors: Kotecki, D.E. Wildman, H.S. Yu, C. Natzle, W. Laibowitz, R.B.

Title: NANO PHASE FABRICATION OF COPPER-GLASS CERAMIC COMPOSITE VIAS IN CORDIERITE SUBSTRATES
10/05/92 Opened as Discl YO8920907 in US Status:Published
10/08/92 Sent to Evaluator
12/17/92 Discl Review Action:Publish
01/06/93 Mailed to Tech Discl Bulletin 09/02/93 Published Pts:1
Co-inventors: Kang, S.K. Shaw, T.M. Brady, M.J.

Title: METHOD OF SINTERING ALUMINUM NITRODE
11/06/92 Opened as Discl FI8920668 in US Status:Closed
11/06/92 Sent to Evaluator
12/18/92 Closed
Co-inventors: Takamori, T. Shinde, S.L.

Title: METHOD OF SINTERING ALUMINUM NITRIDE

11/06/92 Opened as Discl [redacted]8920667 in US Stat[redacted]Closed
11/06/92 Sent to Evaluator
12/18/92 Closed
Co-inventors: Takamori, T. Shinde, S.L.

Title: ALUMINUM NITRIDE BODY AND METHOD FOR FORMING SAID BODY UTILIZING A VITREOUS SINTERING ADDITIVE
08/13/92 Opened as Discl FI8920525 Status:Filed
08/17/92 Sent to Evaluator
09/29/92 Evaluated Action:Search
12/23/92 Discl Review Action:File
05/10/95 Filed as Docket FI992168B in US Rating: 2 Pts:3
05/28/96 Issued as Patent 5520878 in US
Co-inventors: Takamori, T. Shinde, S.L.

Title: ALUMINUM NITRIDE BODY AND METHOD FOR FORMING SAID BODY UTILIZING A VITREOUS SINTERING ADDITIVE
08/13/92 Opened as Discl FI8920525 Status:Filed
08/17/92 Sent to Evaluator
09/29/92 Evaluated Action:Search
12/23/92 Discl Review Action:File
12/22/93 Filed as Docket FI992168A in US Rating: 2 Pts:3
01/09/96 Issued as Patent 5482903 in US
Co-inventors: Takamori, T. Shinde, S.L.

Title: GOLD DOPING OF YBA2CU3O7-8 AS A MEANS OF INCREASING TRANSPORT CRITICAL CURRENT DENSITY
02/12/92 Opened as Discl YO8920161 in US Status:Closed
02/14/92 Sent to Evaluator
05/15/92 Closed
Co-inventors: Daeumling, M. Shaw, T.M.

Title: PROCESS FOR PRODUCING CERAMIC CIRCUIT STRUCTURES HAVING CONDUCTIVE VIAS
07/19/89 Opened as Discl YO8890552 Status:Filed
07/25/89 Sent to Evaluator
08/10/89 Evaluated Action:Search
07/30/90 Discl Review Action:File
12/17/92 Filed as Docket YO990091B in US Rating: 2 Pts:3
08/16/94 Issued as Patent 5337475 in US
Co-inventors: Vallabhaneni, R.V. Giess, E.A. Farooq, S. Cooper, E.I. Kim, Y.H. Vanhise, J.A. Aoude, F.Y. Muller-landau, F. Shaw, R.R. Walker, G.F. Rita, R.A. Neisser, M.O. Park, J.M. Shaw, T.M. Brownlow, J.M. Kim, J. Knickerbocker, S.H.

Title: VIA PASTE COMPOSITIONS AND USE THEREOF TO FORM CONDUCTIVE VIAS IN CIRCUITIZED CERAMIC SUBSTRATES
07/19/89 Opened as Discl YO8890552 Status:Filed
07/25/89 Sent to Evaluator
08/10/89 Evaluated Action:Search
07/30/90 Discl Review Action:File
03/20/91 Filed as Docket YO990091A in US Rating: 2 Pts:3
02/01/94 Issued as Patent 5283104 in US
Co-inventors: Vallabhaneni, R.V. Giess, E.A. Farooq, S. Cooper, E.I. Kim, Y.H. Vanhise, J.A. Aoude, F.Y. Muller-landau, F. Shaw, R.R. Walker, G.F. Rita, R.A. Neisser, M.O. Park, J.M. Shaw, T.M. Brownlow, J.M. Kim, J. Knickerbocker, S.H.

Call your award coordinator, IPL department, or T/L 826-2680 for help.

SEND MAIN MENU OTHER OPTIONS

- T.R. McGuire, A. Gupta, P.R. Duncombe, M. Rupp, J.Z. Sun, R.B. Laibowitz, W.J. Gallagher & G. Xiao "Magnetoresistance and Magnetic Properies of $(La_{1-x})MnO_{3-\delta}$ Thin Films" 3M Conf. Proc: 4/96
- T.R. McGuire, P.R. Duncombe, G.Q. Gong, A. Gupta, X.W. Li & G. Xaio "Magnetoresistance & Magnetic Properties of $(La_{1-x})MnO_{3-\delta}$ (Vacancy) Bulk Materials" 11/96 3M conf CMR Open Forum entry
- J.Z. Sun, L. Krusin-Elbaum, A. Gupta, G. Xiao, P.R. Duncombe, W.J. Gallagher & S. P. Parkin "Magneto-Transport in Doped Manganate Perovkites" 3M conference 11/12-15/96 Atlanta, Georgia
- P. Lecoeur, A. Gupta, P.R. Duncombe, G. Gong & G. Xiao "Emission Studies of the Gas-Phase Oxidation of Mn during Pulsed Laser Deposition Managanates in O2 & N2O Atmospheres" JAP 80(1), 7/1/96
- J.Z. Sun, L. Krusin-Elbaum, A. Gupta, G. Xiao, P.R. Duncombe, W.J. Gallagher & S.S.P. Parkin "Colossal Magnetoresistance in Doped Manganate Perovskites" IBM J&D to appear 1996/97
- A. Gupta, G.Q. Gong, G. Xiao, P.R. Duncombe, P. Trouilloud, P. Lecoeur, Y.Y. Wang, V.P. Dravid, & J.Z. Sun "Grain Boundary Effects on the Magnetoresistance Properties of Perovskite Manganite Films"
- J.Z. Sun, W.J. Gallagher, P.R. Duncombe, L. Krusin-Elbaum, R.A. Altman, A. Gupta, Y. Lu, G.Q. Gong & G. Xaio "Observation of Large Low-field Magnetoresistance in Tri-layer Perpendicular Transport Devices Made Using Doped Manganate Perovskites" to appear Appl. Phys. Lett.
- J.Z. Sun, L. Krusin-Elbaum, P.R. Duncombe, A. Gupta & R. B. Laibowitz "Spin-Polarized Tunneling in Doped Perovskite Manganate Trilayer Junctions" APL submission 11/96
- T.R. McGuire, P.R. Duncombe, C.Q. Gong, A. Gupta, X.W. Li & G. Xiao "Interlayer Exchange Coupling & Magnetoresistance Of LCMO/LSMO 67/33 Multilayers" APL submission
- R.B. Laibowitz, T.M. Shaw, D.E. Kotecki, S. Tiwari, A. Gupta, A. Grill, & P.R. Duncombe "Properties and Applications of Thin Films of Lead Lanthanum Titanate (PLT) and Barium Strontium Titanate (BST) APS mtg 3/18-22/96
- P.R. Duncombe. S.L. Shinde, & T. Takamori "Aluminum Nitride Body Utilizing A Vitreous Sintering Additive" US05482903 1/9/96 (EF Plaque)
- P.R. Duncombe, S.L. Shinde, & T. Takamori "Aluminum Nitride Body & Method for Forming Said Body Utilizing a Vitreous Sintering Additive" US05520878 issued 5/28/96; I.A. Patent issue Award: 8/96
- Ali Afzali-Ardakani, Mike Brady, Dah-Weih Duan, Peter Duncombe, Chris Feild, and Paul Moskowitz "RF Transponder for Metallic Surfaces" Docket#:YO895-0329 submitted: 8/2/95
- D.E. Kotecki, R.B. Laibowitz, W. Natzle, C. Yu, H. Wildman, P.R. Duncombe "Method for Cleaning the Surface of BST Prior to Electrode Deposition" Application #:FI996047 draft #1 under review
- E.I. Cooper, P.R. Duncombe, R.B. Laibowitz, "Peroxide Etchant Process for Titanate Dielectrics" Docket: YO895-0434 rated file; in prep.
- D.A. Neumayer, P.R. Duncombe, R.B. Laibowitz, & A. Grill "Sol-Gel Processing of BaSrTiO3 Films" submitted to International Symposium on Integrated Ferroelectrics (ISIF: 3/2-5/97) Santa Fe, N.M.
- A. Grill, R. Laibowitz, D. Beach, D. Neumayer & P.R. Duncombe "Effect of Base Electrode on the Crystallization & Electrical Properties of PLT" IBM RC 20402 (90185) 3/5/96
- D.A. Neumayer, P.R. Duncombe, R.B. Laibowitz & A. Grill "Effect of TiOx Nucleation Layer on Crystallization of Sol-Gel Derived Bi4Ti3O12 Films" ISIF submission 3/97
- C.D. Dimitrakopoulos, P.R. Duncombe, B.K. Furman, R.B. Laibowitz, D. Neumayer, S. Purushothaman, J. Shaw "Field Effect Transistor for Low Voltage Operation" Disclosure YO896-0358 rated file: 9/11/96
- R.B. Laibowitz, P.R. Duncombe, D. Neumayer,K.L. Saenger, A.G. Schrott "Noble Metal Surfaces" YO896-04xx rated "file" 10/96
- T. Shaw, R.B. Laibowitz, P.R. Duncombe & A. Gupta "High Dielectric Constant Barium Lanthanum Titanate-Based DRAM Structures" Disclosure #: YO898-0681 rated File 5/96 in preparation
- D. Neumayer, P.R. Duncombe "Fabrication of Barium Strontium Titanate Films" YO896-04xx rated File 10/96 in preparation

IBM Commitments: **To Win** **To Execute** **To Teamwork**

ATTACHMENT B

IBM

T01001

Technical Notebook

Book IV

User's Initials and Last Name:		
P DUNCOMBE		
Employee Serial:	Date of First Entry:	Date of Last Entry:
155139		

Security Classification:

11/12/87 6/88

11/12

70/30 - 25-25 } C1GEP2 - ~12:30 start

3.094 ?	0.574	0.179		4.07	"63.9" 123 basis ~~87.6~~ !?
	1.458	0.455	0.760		
~4hrs					
3.047	0.515	0.158		5.65	88.7 better
≤~1.5% loss	1.308	0.401	0.539		p1 → 83.4

11/12

$SrTiO_3$ - ST3 ⇒ 32 hrs ST2 pcs. ⇒ 48

11/13 $SrTiO_3$ ⇒ ST3 ⇒ cooled in morning see book III, pg (A7)

4.024	0.510 ✓	0.240 ✓		~5.01	"1.04(2) % dense"
no wgt loss	1.295	0.610	0.803	same	fully

~48hrs (+ cooling 3 mornings, stepwise) sintering pellet

Cutting record

start 0.425 (0)

+ 0.060 Δ - saw(0.015) = 0.045 ~ 1.14mm w/ flattening ~ 1mm ✓

.485 (Δ 0 ↳ 0.042 (1.08 mm) OK

0.060

0.545 (0.045 Δ resid.; actual ⇒ 0.052 → (1.32)mm

(55) .0465 (1.18mm

0.0523

bottom (0.6 - 0.69) not flat (1.52 mm)

10/13

2

PRE

ST4 - some deformity on 1 side (a shim worthy) Way final piece of ST2 @ 4:55

4.178	0.584	~0.287		3.316	68.9
	1.483	0.729	1.26		~average

will remove monday morn:. ~ 6!-6 ~~SAT~~, 6SAT-6SUN, ~63+ hours projected

10/16

4.169	0.510	~0.250		4.98	1.035 <CONSISTENT>
(0.2 %)	1.295	0.635	0.837		

G1 ISO-26,000 OXL-3300

4.01	0.578	0.248	~~0.542~~	~~3.796~~
	1.468	0.630	~~0.[illegible]~~	
			1.066	

density est. (figure 65%) => 5.785 RANGE (5.37 - 6.27)

C1ZT1

10/17 3300/26,000 EXTREMELY SHINY, flat surfaced, ~~wall~~ sharp convoved pellet

3.105	0.566	0.193(4)		3.90	61.2%
	1.438	0.490	0.796		

G1 16hrs

3.985	0.578	0.250	(no change)	3.738	(lost 0.5% density)

10:18 IN hot furnace, packed T_c - 520C

	T_c	T_s	T_{SET}	
20	977	745	971	to 'push' ΔT
21	↓	838		
22		898		
23		935		23 1/2 => 951
24		949	956	~SET-PT.
2:23				"OFF" for slo-cool (first stepped to 840)
2:54	435			

The above understood Date and Date

10/17

C1 2 f 1 → pellet multiply cracked as if organic residue vaporized, evidence of vapor transport to support plate, etc.. ~~ex~~ Nat

2.925 Δ 0.18
(5.5%)

9.79 - 3.105 => 6.685

10/18

G1 - post 4.044 split in 4 pieces (seemingly on cooling)

G2 A.1	0.579	0.253		3.75 ✓	pellet slightly disfigured, but etc.
	1.471	0.643	1.093		
33					
	4.155				
	0.510	0.220		5.64	about expected density
	1.295	0.559	0.736		

D.D.1 Pre
3.10 0.5765" 0.191"

[illegible]	0.513	0.165		5.61	88.2
	1.303	0.419	0.559		

5.75

4
11/24

Thermodyne Tube furnace set-up specs -

thermocouple: dia. ~0.255 length 20" + → USED 23"

Set-up complete w/ plug in jacks, incl. WIRE, 5 couples.

11/30 Analytical Submission

C1 -	0.75 g	$Y_{0.02}Ba_{0.98}Cu_{0.6}$	Y, Ba, Cu
C2 -	1.1	Y_2O_3	Y, trace 99%
C3 -	2.0	"BaO"	Ba,
C4 -	1.0	TiO_2	Ti, trace
C5 -	2.0	$SrTiO_3$ pre ↓	Sr, Ti, trace ↓
C6 -	1.0	post mill	
C7	2.0	DRC 123.	Y, Ba, Cu
C8		DD 123	Y, Ba, Cu
C9		off comp 2:11	

The above understood and witnessed by _____ Date _____ and by _____ Date _____

'New' ~~30~~ 20.25 g GRINDING CHARGE of $SrTiO_3$ IN MILL (3:10) off (4:17)

O_2, compressed AIR, CO_2 cylinders obtained w/ regulators
Ar

YIELD ⇒ 20.4g ∴ MUST BE some from old batch or ZrO_2

COMBINED W/ OLD PWDR ⇒ 23g of milled powder

12/2 C1. batch 45.6 grams left
39.56 g (~6 g kept for files
10.5-
29.5 left for pellets
~10 for grinding charge TFE/Toluene

NEW BOTTLES ORDERED, NO TEFLON AVAILABLE, - approx - 60 hrs total

$SrTiO_3$ pellets ⇒ 10-10 (29) hrs Tues down N4 ∴ 2-2am Wed (~12?)/N12-12 Thurs (24)

ST4, ST6 - start 10A.M. 12/9, numerous interuptions due to furnace malfunctions, out 12:00 P.M. 12/10

ST5 edge chips 1 side OK otherwise 21 ISO.

*'4.08	0.285	0.584 0.52			
(4.01)	0.237 0.602	0.520 1.321	0.825	4.94	1.027

ST6 large chip during iso pressing in 3/4 side, must do

*4.128	0.586	0.886			
4.15	0.513 1.303	0.249 0.632(5)	0.843	4.92	1.023

*'between cracks

950C RUN

C1P9

GREEN	3.108	0.577	0.185		3.92	61.5
		1.466	0.470	0.793		
POST	3.1	0.514	0.161		5.66	88.9%
	no loss	1.306	0.409	0.548		
GREEN	DRC2	0.579	0.177		4.19	65.8
	3.204	1.471	0.450	0.765		
POST	3.2	0.551	0.165		4.96	77.9
	no loss	1.400	0.419	0.645		

12/7 pellets not in best shape after iso at 26000

975C RUN

	C1P10	0.574	0.185		3.99	61.9
	3.090	1.458	0.470	0.785		
	3.056					
POST	1% loss	0.508	0.157		5.88	92.3 → PROBABLY (93) +3.4%
		1.288	0.399	0.579		
	*DRC3	0.579	0.181		4.24	66.6% ~
	3.318	1.471	0.460	0.782		
POST	crack still apparent, but holding					
	3.293	0.547	0.168	0.647	5.08	(79.9) + 2%
	0.75% loss	1.389	0.427			

C1P10 Pynometer 91.8 ⇝ 92 ∴ MOSTLY closed porosity (95% of our pure density basis)

* cracked in ½, but holding. Will go in { see if it heats.
To TEMP @ 4:05 ⇒ 2 HRS 6:05 START RAMP down
9:00 A.M 12/8 start cooling, OK NOT FLOWING WHEN ARRIVED, THOUGH COULD HAVE HAD BACK PRESSURE

12-8

100% E/G mix ⇒ new wght calc,

(4.0 g) E basis (transfered to jar for physical mixing)

92.0913 g/mM cation ∴ 4.0/92.0913 = 0.0434 mM

0.0434 mM is BASIS for mix of 0.7 mM Eutectic

0.0434/0.7 = 0.0620 mM total ∴ 0.3 mM B11

0.3 (0.0620) = 0.0186 mM (94.6725 g/mM 211) = 1.761 g 211

1.761 g 211
4.0 g E
5.761 g mix

tare 0.83 — 5.76 — 5.68 recovered — .08 g loss on mixing — 5.53 after grinding (slight loss on transfer)

1 pellet pressed ⇒ EG1 ⇒ to temp 12/10 @ 3:40-45 PROJ OUT TO BE 5:45

2.57 — 0.580 — 0.153 — 3.88 — "60.9" ⇒ 74%?
1.473 — 0.389 — 0.663

Rel. density calc 0.3 (6.00) + 0.7 (4.9) ⇒ 5.23 approx theoretical
(6.00 ← EMPIRICAL D's; 4.9 ← D's)

2.543 (1% wght loss) — ~0.611 — 0.161 — 2.825
1.55 — 0.480 — 0.90

Restarted for overnite RUN

The above understood and witnessed by ____ Date ____ and by ____ Date ____

12/9

8

IBM Technical Notebook

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

B_2C_5O

SAMPLE I.D. B660 eutectic
SOURCE D.Garland
TOTAL WEIGHT 18.855 g.
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 14.794 g.
DATE
OPERATOR PRD
OUTGASSING CONDITIONS
ADDED VOLUME, V_A cc
CELL HOLDER VOLUME, V_c cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

R=3.687

	RUN 1	RUN 2 (3.688)	RUN 3
P_2	18.362	18.488	
P_3	4.980	5.013	
V_p	3.023 cc	3.034 cc	cc
DENSITY	4.89 g/cc	4.88 g/cc	g/cc

± 0.24 (5%)
4.65 → 5.13
{permeance / limitation}

16

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

B-Oleic
SAMPLE I.D. O_2-N_2O_2
SOURCE Sumitomo
TOTAL WEIGHT 12.606 PRE
TARE WEIGHT 9.006 g.
SAMPLE WEIGHT 8.546 g.
DATE 12-9-87
OPERATOR PRD
OUTGASSING CONDITIONS
ADDED VOLUME, V_A 26.52 cc
CELL HOLDER VOLUME, V_c 34.85 cc

POST 9.262 − 4.062 = 5.15

13.867 − 4.061 = 9.806

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

	POST − SUMITOMO	
	V_c 34.85	V_c 34.85
	R=3.687	R=3.653
P_2	18.501	18.424
P_3	5.087	5.079
V_p	2.419	2.395
D	4.05 (±2%)	4.09 (±3%)

DATA

	PRE (a.s.i.) RUN 1	POST RUN 2 R=3.549	RUN 3 R=3.549
P_2	18.320	18.502	18.534
P_3	5.078	5.213	5.222
V_p	— cc	1.296 cc	cc
DENSITY	4.15 g/cc	3.96 g/cc	g/cc

3.96 → 5.04

Rec'd 01

16

III. DENSITY WORKSHEET

best guess - 5.9
STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 211
SOURCE PRD
TOTAL WEIGHT 19.662 g.
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 15.601 g.
DATE 12-9-87
OPERATOR PRD
OUTGASSING CONDITIONS
ADDED VOLUME, V_A cc
CELL HOLDER VOLUME, V_c cc

RUN2
20.477 − 4.061 = 16.416

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

R=3.65

	RUN 1	RUN 4 R=3.651	RUN 7 R=3.663	R=3.664
P_2	18.557	18.568	18.564	18.529
P_3	5.084	5.085	5.052	5.098
V_p	2.578 cc	cc	2.736 cc	2.748
DENSITY	6.05 g/cc	g/cc	6.000 g/cc	5.97

± 0.3
(5.95 - 6.35)
*Ave Run 1 & 2 = 6.00
Actually 6.05 (rounded)

16

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123
SOURCE DRC
TOTAL WEIGHT 21.026 g.
TARE WEIGHT 4.063 g.
SAMPLE WEIGHT 16.963 g.
DATE 12-9-87
OPERATOR PRD
OUTGASSING CONDITIONS
ADDED VOLUME, V_A 34.85 cc
CELL HOLDER VOLUME, V_c 25.52 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

DATA

R=3.6625

	RUN 1	RUN 2 R=3.668	RUN 3
P_2	18.598	18.596	
P_3	5.078	5.078	
V_p	2.73 cc	cc	cc
DENSITY	6.21 g/cc	g/cc	g/cc

16

12/9

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123-3um DATE 12-9
SOURCE DIMNS OPERATOR PRD
TOTAL WEIGHT 19.700 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 15.639 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R=3.646

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.603	18.561	~~18.504~~
P_3	5.103	5.091	~~5.082~~
V_p	2.523 cc	cc	cc
DENSITY	6.199 g/cc	g/cc	g/cc

no purge

16

III. DENSITY WORKSHEET

STEREOPYCNOMETER RUN 2
TRUE POWDER DENSITY

SAMPLE I.D. 123 DATE 12-9
SOURCE C1 OPERATOR PRD
TOTAL WEIGHT 22.944 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 18.883 g. ADDED VOLUME, V_A 34.85 cc
CELL HOLDER VOLUME, V_c 85.52 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R→ 3.692

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.508		
P_3	5.014		
V_p	3.082 cc	cc	cc
DENSITY	6.13 g/cc	g/cc	g/cc

16

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 123 DATE 12-9-87
SOURCE C1 - 3um OPERATOR PRD
TOTAL WEIGHT 14.459 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.063 g.
SAMPLE WEIGHT 10.396 g. ADDED VOLUME, V_A 84.57 cc
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R=3.58

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.677	18.643	
P_3	5.218	5.208	
V_p	1.703 cc	cc	cc
DENSITY	6.10 g/cc	6.105 g/cc	g/cc

LOT BACK TO ZERO

16

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. C1P10 DATE 12-10-87
SOURCE C1-975248 OPERATOR PRD
TOTAL WEIGHT 6.613 g. OUTGASSING CONDITIONS
TARE WEIGHT 4.061 g.
SAMPLE WEIGHT 2.552 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_c 34.85cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

R= 3485

DATA

	RUN 1	RUN 2	RUN 3
P_2	18.148	18.182	
P_3	5.208	5.217	
V_p	0.4355 cc	cc	cc
DENSITY	5.8599 g/cc	g/cc	g/cc

over 615 → 95.6
637 → 91.8 (92)

16

→ 4.69 + 58.715

10 12/10

IBM Technical Notebook

PWDRS for Analysis ⇒ NOTE ENTERED @ CONF. TIME 12/11, SENSITIVITIES NOT ENOUGH Need to increase by 10X at least.

	Species	Description
~~E1~~ -	Y_2O_3	left exposed to air
	TiO_2	
C1	YBa_2Cu_3	
DD1		
DRC		
PII		off comp
EI	$Y_{0.02}Ba_{0.33}Cu_{1.6}$	off comp

Table I - Precision[1] of Metals determined by ICP in $La_{1.6}Sr_{0.2}CuO_4$ and $YBa_2Cu_3O_7$ Thin Films.

Element	x[2]	S.D.	R.S.D. (%)
La	1.80	0.08	4.64
Sr	0.20	0.01	5.52
Cu	1.00	0.14	3.52
Y	1.00	0.05	5.60
Ba	2.04	0.07	3.43
Cu	3.00	0.11	3.67

[1]Based on 7 determinations

[2]Calculated atomic ratios

Spec 123

Y (1.000) ⇒ ± 0.019 0.314 - 0.352

Ba (0.667) ⇒ ± 0.023 0.644 - 3.690

Cu (1.000) ⇒ ± 0.036? 0.963 - 1.036?

Theoretical wgt % calcs.

Anal 1

TiO_2 ⇒ 47.90/79.8988 ⇒ 59.95 57.3 ERROR reported.

$SrCO_3$ ⇒ 87.62/147.62935 ⇒ 59.35 NOT ANALYZED

$BaCO_3$ ⇒ 137.34/197.34935 ⇒ 69.59(2)

BaO ⇒ 89.566 88.9 99.26 !

$SrTiO_3$ ⇒ Sr ⇒ 47.74(5) M.W. 183.5182
Ti ⇒ 26.10(1)

C5- 22.2 Ti 85.05% (15% poor)
49.4 Sr "3.48% rich"

C6 24.2 Ti 92.72 (7.3% poor)
50.6 Sr "5.98% rich"

86.5
92.5

12/12/14 both well shaped pellets

C1P11 - 15026

3.673	0.574	0.215		4.03	63.3
	1.458	0.546	0.9116		

92+ final from T.SHAW

C1fp(#)? 15026

3.058	0.561	0.206		3.606	56.6 as usual
	1.437	0.523	0.848		

- final microstruct full of liquid, bimodal g.s. { cracking
- no final density recorded

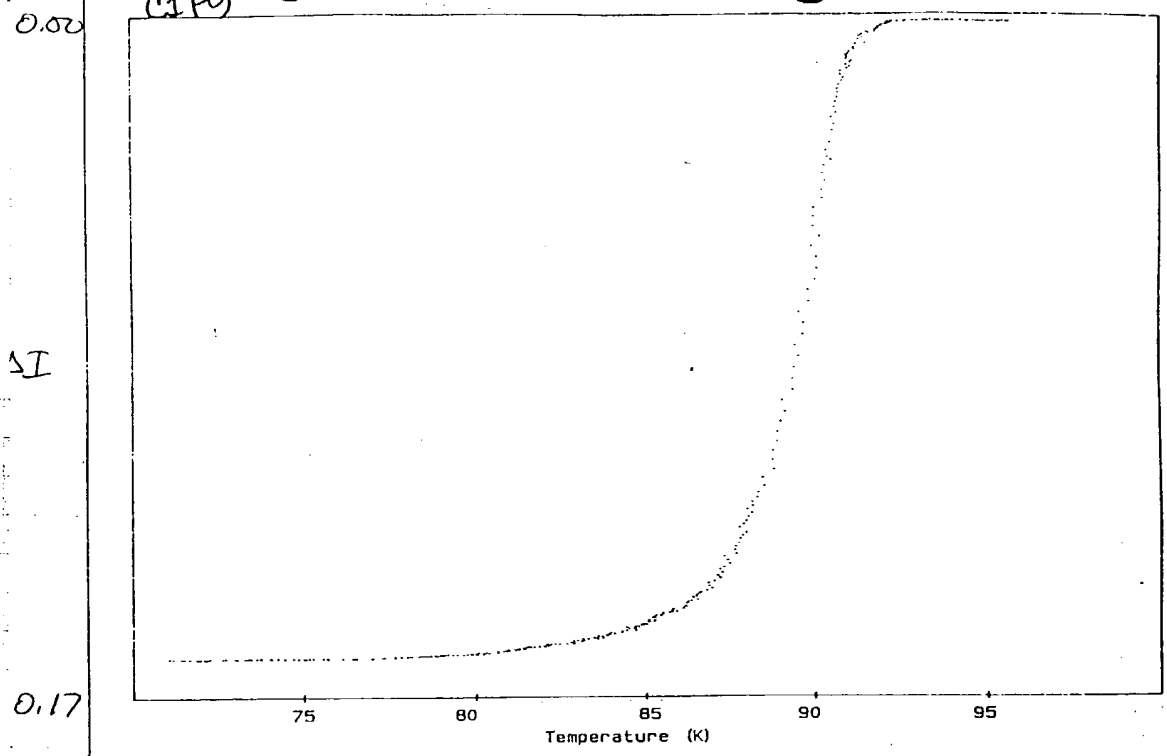
12/16 pellets in furnace from 12/15 in expurge.
To temp (10°C/min ramp from RT) @ 10:50 A.M.
Low (leading) side undershoot 974, high (downside) overshoot 978.
Stable valuation 974-976

start ramp down 1:00 (12:50) p.M. (to 600C where SOAK for 48 HOURS)

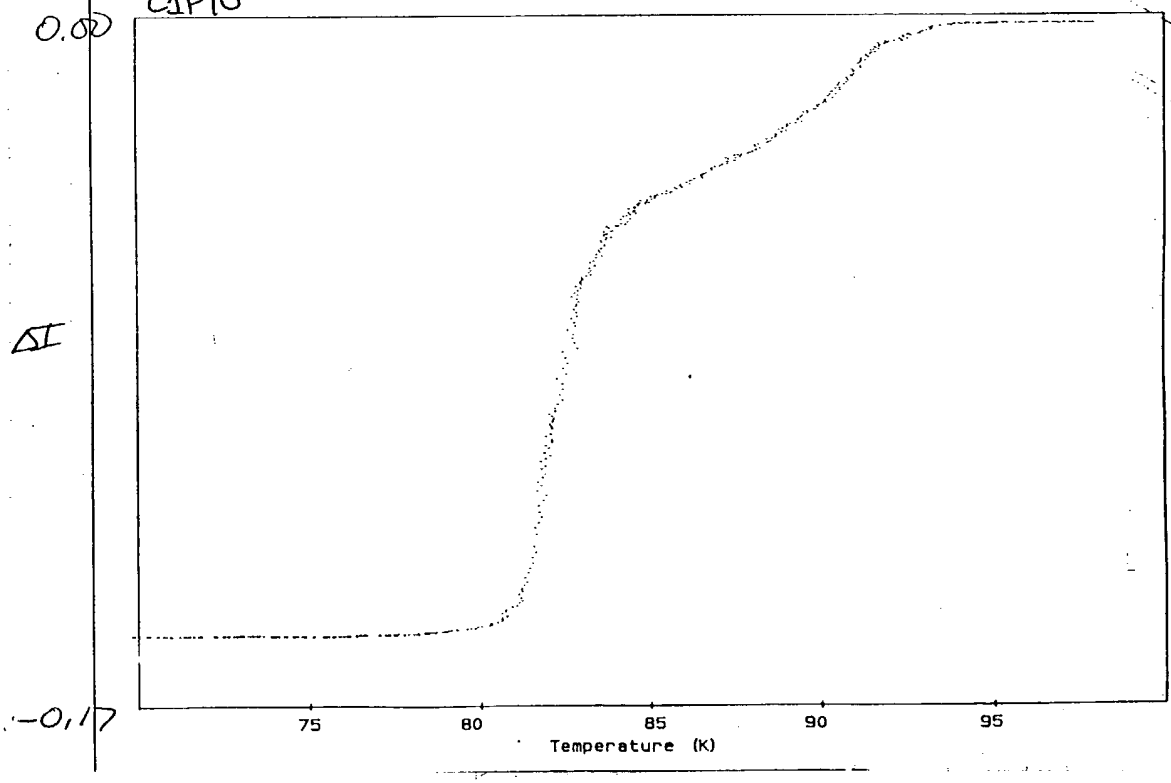
{ Dif coef: $2 \times 10^{-15} m^2/s^{-1} \times 2 \times 10^{-15} \frac{m^2}{s^{-1}} \times \frac{ft^2}{(0.3048 m)^2} = 2.153 \times 10^{-14} ft^2/s$ }

The above understood and witnessed by _____ Date _____ and by _____ Date _____

C1P9



C1P10



C2 Batch → $Y_1Ba_2Cu_3O_7$ 200g

From C1 batch calc. (pg. 34 Book III → 72 Book II)

	wt Frac.	Purity	Mult.	Final
Y_2O_3 ⇒	17.1535 ⇒	17.1707 ⇒	×2	34.34
$BaCO_3$ ⇒	46.5934	100 ⇒	×2	93.1868
CuO ⇒	36.25(81 ⇒	36.2893(1) ⇒	×2	72.57(9)

$BaCO_3$ conversion: $93.1868 \frac{197.35}{153.34} = 119.932(3) \div 0.99 \Rightarrow 121.14(4)$

O.K. everything is Ba rich by analysis, so obviously not correct → 119.93 ± .606 → 120.54

Apply ½.

$BaCO_3$

tare: 279.67 + 120.54 = 400.21 won't read, but will tare

reads: 120.57(4-6) was 4/5

CuO

tare: 0.8717
reads 72.58 transferred quant. tare to zero w/ paper

Y_2O_3 transferred quant

34.34/5 paper weighs -0.1 after checked due to static glove charge, but after glove/charge removal 0.00. Think OK since cat-spilled glove also (not more than 0.3% error)

Expected total right } 227.46 g dry

BaO - 5.72 g/cc $BaCO_3$ - 4.43 Y_2O_3 - 5.01 CuO - 6.3-6.49

∴ if bumping occurs w/ selective loss, $BaCO_3$ should preferentially be lost if not ~~well~~ uniformly suspended.

The above understood and witnessed by Date and by Date

Except for 1 bump (0.06 g recovered) ⇒ very smooth, uneventful preparation. Placed in drying oven for weekend drying. (oven cleaned before use also)

12/21 after breaking up coke and re-baking under vac @ 70C for 3+ hrs.

~~CRUX~~ #1 transferal ideally want 75 per crux

166.67 → 0.97
tare 86.21
80.46 g "recovery"
-.01

totals
80.46
77.74
68.29
226.49 expected 227.46 (99.57%)

0.3 g recovered on ~~bumping~~ bake.

226.79 total 99.7 %
227.46

CRUX 2 172.72
94.98
77.74
+.03 "recovery"

CRUX 3 173.46
105.17 "white"
68.29

RXN. RUN 1 ⇒ IN { on @ 4:00 PM 320 RAMP to 940C, 450 cool RAMP
14 hrs + 3 up + 2 down = 20 hrs total

12/22 ...

{12/21}

12/17-18 CENTURR $SrTiO_3$ RUN

Δ 500 psi

2100 - 1700 psi Ar usage 16 HR SOAK @ 1800 w/ 16.7 up/400 hour ramp

Running Si-Christy run Prog. 05

∴ for 12/21 ⇒ ⊗ $SrTiO_3$ RUN 24 hrs ⇒ 1000 psi

16

40 hrs ⇒ 1500 psi max permissable

Set for 36 ⇒ RAMP STARTED @ 1:25 p.m. 12/21

3 hrs to ramp

4:25

36 hours SOAK

40:25

4.25

1600 psi @ 300C RAMP up. 44.5 hours total should be O.K

(3 HRS ⇒ 1000) ⇒ 15,000 projected usage.

12/22 9:00 AM

19.3 soak hours left ∴ Δt ⇒ 16.7 + 3 ⇒ 19.7 {(16 - 11250) psi ⇒ 4,750

∴ 241.1 psi/hr. 19.3 + 4.25 = 23.55 (241.1 psi/hr) = 5,680

11250 - 5680 = REMAINDER of 5,572 psi } could run longer if rate remains constant

6:00 pm

11,250 - 9,000 ⇒ 2,250/(16.7 - 10.2) = 2250/6.5 = 346.2 !

346.2 (10.2 + 4.25) = 65,000 psi + (9000) = 4,000 to spare ✓

12-22

C2 RXN SEEMS GOOD, NO APPARENT LIQUID, LARGE SHRINKAGE NO VISIBLE GREEN, GOOD BLACK COLOR, BEFORE UNLOADING.

~~Post wght.~~

CRUX #1
initial
tare

166.97
86.21
80.76

100% EXPECTED RXN weight.

80.76 + (80.76 × (−0.1182)) = 71.214

loss calc.

227.46 theoretical powder

$\frac{120.54}{227.46} = 0.52994$ wgt % $BaCO_3$

CRUX #2
initial

172.72
94.98
77.74

as above = 68.551

$\frac{153.34}{197.35}(0.52994) = 0.41176$

$\Delta = 0.52994 - 0.41176 = 0.1182\%$ total

WRONG?

CRUX #3

173.46
105.17
68.29

as above = 60.218

$\frac{60.218}{199.983 \text{ total}} / 0.997 = 200.58$ ✓✓ OK.

Actual yields - 14 HR RXN @ 940C

		total wgt initial tare	ABOVE EXPECTED WEIGHT,	% RXN
CRUX #2 2.27g wght loss	170.45 ~~94.98~~ ~~75.47~~	75.47	6.919 (9.589)	24.7
CRUX #1 ~~2.05~~ wght loss	169.92 86.21 78.71	78.71	7.496 (9.546)	21.5
CRUX #3 1.55g wght loss	171.91 105.17 66.74	66.74	6.522 (8.072)	19.2

CONSISTENT

21.8% ave. O.K.

The above understood and witnessed by

Date

Date

18 Recheck of wght loss calc.

Ba 137.34
O 15.9994
153.3394 ~~153.34~~ 1C
~153.34

Ba 137.34
3Os 47.9982
12.0115
197.34935
~197.35

$\frac{153.34}{197.35} = 0.776995\ (120.54) = 93.658999?\ \Delta 26.881\ g$

26.881 g / 3 cruc. = ~8.96 g/crucible ~ correct

Individual wght inquiries during grinding 12/29

crux 3 66.72 unloaded

105.17 tare
171.82 loaded
171.91 previously
.09 g loss = 0.135%
66.74

169.38
171.82
-2.44 loss
+ -1.55
3.99

crux ~~2~~ 1
86.20 tare (0.19/20)
78.75 load (-0.02 ~~78.73~~
78.69 gain after grinding
.06 g loss > 0.076% loss
164.85
78.65 0.09 g loss = 0.115% los

161.68
164.85
-3.17 loss
+ -2.27
5.44 total to date

crux ~~1~~ 2
75.49/8 unloaded
94.99/8 tare
75.46 load (pre crux)
170.44 loaded.
75.46 0.03 loss

167.18
170.44
-3.26 loss
2.05
5.31

The above understood and witnessed by ____ Date ____ and by ____ Date ____

IBM Technical Notebook

Samples were incompletely converted, as right loss indicated. Top was black, but went through a transition of greens progressly lower w crucible. Grnd powder was a dull forest green. Crux 1 slightly darker than 2 & 3. All had white hard oxyhydrates (presumably $Bg(OH)_2$). Tops inhibited oxygen flow throughout crucible. No tops used for second RXN. Heat treatment

12/29 New losses consistent w/ crux loading. Conversion now up to 70.8 ~ 71%. Will regrind and ~~re~~ in the weighing loss

Crux 2 unloaded	95.02				
ground	72.10				
crux 1	86.24 ✓				
grd.	75.40				
crux 3	105.19				
grd pot	64.15				
crux 1 & 3 (3 crucibles)	192.24 86.29/1	188.21	-4.03	22.82 expected 26.807	(85.1%)
crux 2	199.53 95.01	195.48	-4.05		

12/4 Reground, to 1 crucible

286.06
86.27
199.73 of 202.47 to start (before grd)

∴ (202.47 - 199.73) = 2.74 g loss (1.35%)

20 123

Wght Loss Summary (by run { crucible)

Crucible #	Initial	Post (1)	Post (2)	Post (3)	Crucible
1	80.76	78.71	75.48	102.0	1 & 3
2	77.74	75.47	72.20	100.47	2
3	68.29	66.74	64.21	202.47	
	226.79	220.92	211.89		
theoret	227.96				

Final grd into 1 crucible: pre grnd 202.47
post 199.73
loss 2.74g (1.35% ⇒ 1g spill of sieved prod)

Was 85% reacted before this run.
Total loss so far slightly less than 24.32g/27.46 (88.5-85.%)
Expect less than, but approx. 3.0g loss for complete rxn.
0.52494% Bi_2O_3 { (0.76895% of Bi_2O_3 is Bi_2O)
Look for 283g total upon cooling!

1/5

initial wght.	286.00	199.73	with unloader
post	284.17	197.59	
	001.83g	2.14	

1/4 $SrTiO_3$ synthesis II references book III pgs. 77, A3

TiO_2 - 79.8988 g/M

$SrCO_3$ - 147.6235

$SrTiO_3$ - 183.5182

SrO - 103.6194

Ti - 47.90 IN $SrTiO_3$ 26.1009

Sr - 87.62 IN 47.7446

Take transferred AMOUNT TO SHAKER JAR ($SrCO_3$) as basis for TiO_2 addition

~~201.66 g base~~ $\frac{98.50}{147.6235}$ (0.3286 moles) × 79.8988 g/M TiO_2 = 26.2498
= 26.2547

~~H0.86~~

251.07
202.56
~~48.50~~

251.07 251.07
26.25 /.999 = 26.2763
277.32 target 277.35 actual 277.35/6
46

The above understood and witnessed by _____ Date _____ and by _____ Date _____

COMPS SCRIPT A1 dated 87/12/02 14:32:25 ..... Page 1

Date: 2 December 1987, 13:24:31 EST
From: PLECHAT at YKTVHZ
To: PRD

The laboratory results on your samples are:

# C1	Y	Ba	Cu	O	Cu=1,	ICP
	0.03	0.68		X		
# C2	Y ... 78.1 % (W/W)					
# C3	Ba ... 88.9 % -					
# C4	Ti ... 57.3 % - / error due to static electr. during weighing of sample!/					
# C5	Ti ... 22.2 % -					
	Sr ... 49.4 % -					
# C6	Ti ... 24.2 % -					
	Sr ... 50.6 % -					
# C7	Y	Ba	Cu	O	Cu=1	
	0.34	0.71		X		
# C8	Y	Ba	Cu	O	-	
	0.34	0.71		X		
# C9	Y	Ba	Cu	O	-	
	2.37	1.10		X		

MMP

Date: 21 October 1987, 10:45:18 EDT
From: PLECHAT at YKTVHZ
To: PRD

The laboratory results on your samples are:

# C1	Y	Ba	Cu	O	Cu=1,	ICP
	0.35	0.72		X		
# C1f	Y	Ba	Cu	O	-	-
	0.33	0.70		X		
# C5	Y	Ba	Cu	O	-	-
	2.21	1.06		X		

Other results to follow from Olson,
MMP
Note: I have produced a light green compound from 123 with the formula: Y Ba Cu O . If interested get in touch with me.
12 3 X

---

theoretical

T_1 consistently low 26.1

Sr consistently high 47.7

41.21	initial	197.59	Post
283.17		196.56	196.56
tare 86.61			194.16
196.56		1.03 g lost during grinding	−2.40

Lost another 2.4 g. Must be totally converted @ Thispant.

uniaxial - 7,000/0.371 = 18,870 PSI

0.126	0.611	2.53	4.19	65.8	
0.320	1.55		0.604		

C2P1 green ✓ not final dens. data

C2P2 ⇒ Will leave notes on push later. too busy. Still see no. on crucible however, disheartening.

↳ 1 mill 4.2 μm PSD 10-1 ~ flat dist.

green 27 isopressed

3.59	0.580	0.194		4.274	67.1% (high vs C1)
	1.473	0.493	0.84		

C2P3 - (Green) mill 2 2.53 μm ave., much better behaved pellet

3.55	0.576	0.210		3.96	62.2% (good agreement w/ C1 ✓)
	1.163	0.533	0.896		

C2P2 - removed @ 600°C ⇒ 20°/up to 800, 10°/to 975, 20° down

3.59	0.554	0.186		4.89	76.8% ! terrible slightly higher dwell pellet attrition
	1.407	0.472	0.734		

1/13

C2P3 3.57	0.517	0.185		5.57	87.4%
	1.313	0.47	0.636		

~88

IBM Technical Notebook

1/13

1 transient .996 peak

C2P4 3,775 uni/26,000 psi 988 2HRS/600 over

3.50/1	0.576	0.206		3.787	62.6% consistent
	1.463	0.523	0.89		
1/14 3.49	0.515	0.180		5.68	89.2
	1.308	0.457	0.614		

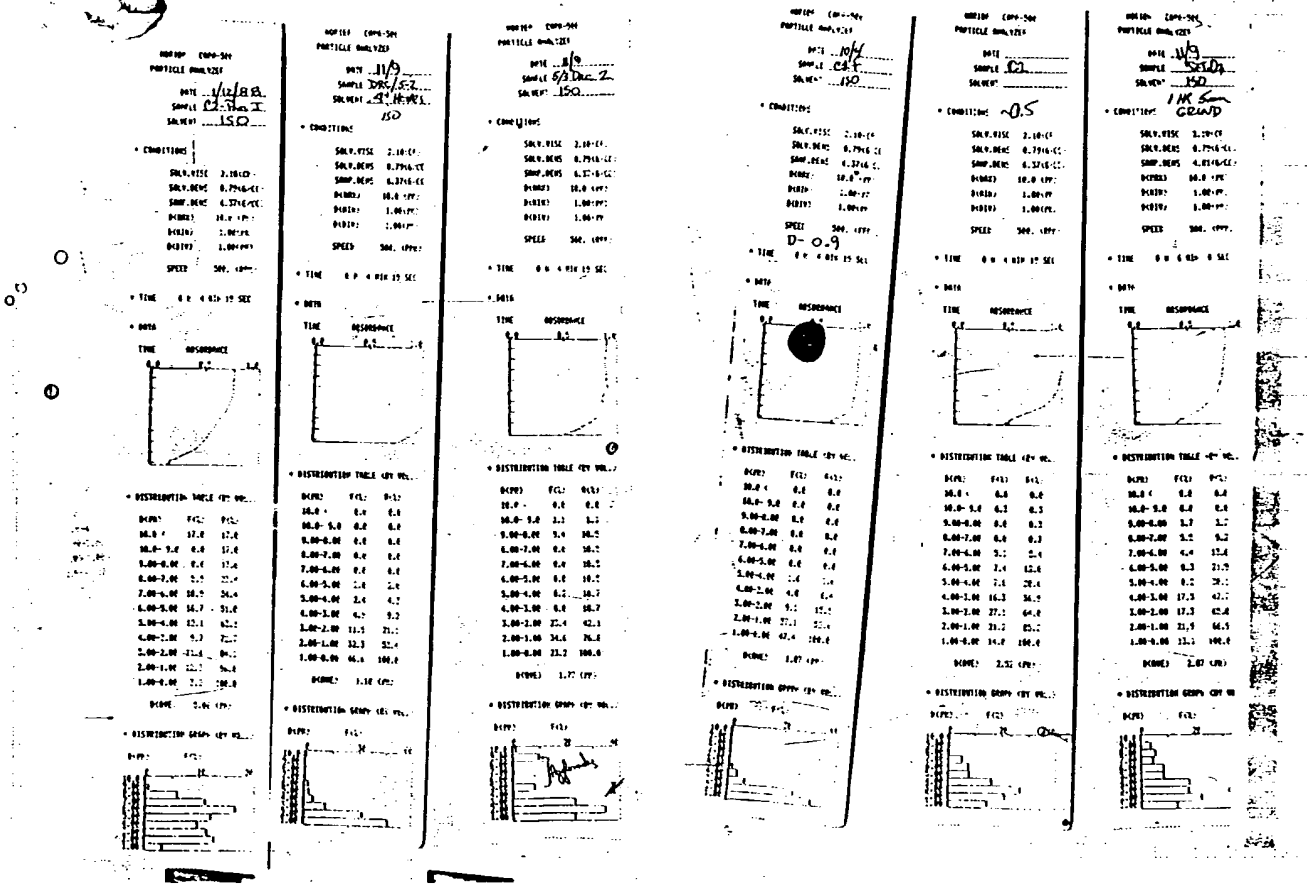
C2P5 3775/24 990

3.18	0.577	0.199		3.74	58.7%
	1.466	0.505	0.85		
3.15	0.496	0.168		5.92	92.9
	1.26	0.427	0.532		

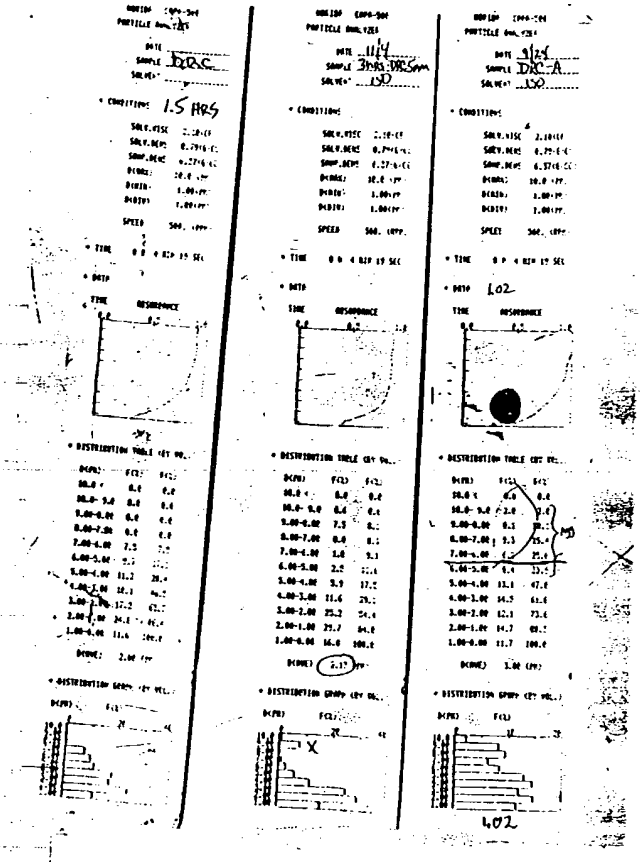
pellet has stress cracking and phomo microstructure with large grain interior and peripheral eggshell of small grains.

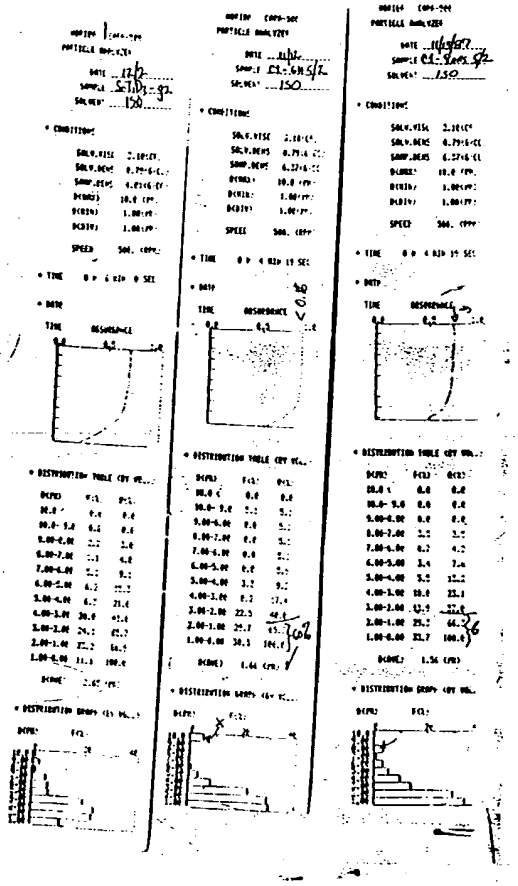
The above understood and witnessed by _____ Date _____ and by _____ Date _____

PSD SHEETS

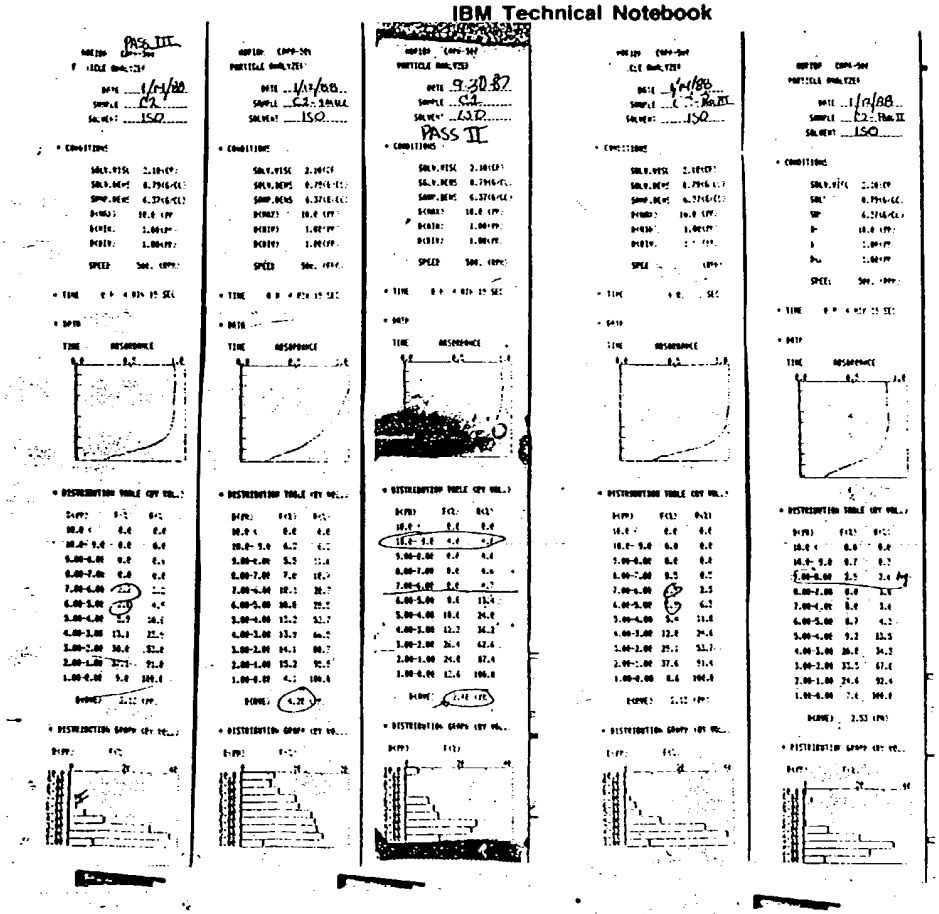


see pg 27 for REST





The above understood and witnessed by _____ Date _____ and by _____ Date _____



The above understood and witnessed by

Date

and by

Date

28 1/A (18,17,16,15 26lb/sq) NOTE ⇒ C1 powder

C1P12, 13, 14, 15 3775/26,5—

C1P12

Grn 3.04	0.574	0.178		3.92	61.5%
	1.478	0.452	0.7755		
3.01	0.506	0.153		5.966	93.66%
	1.285	0.389	0.504(5)		

C1P13

Grn 3.00	0.574	0.175		3.93(4)	61.8%
	1.478	0.444(5)	0.7626		
2.97	0.506	0.150		6.01	94.35
	1.285	0.381	0.494		

C1P14 (*)

Grn 2.89	0.574	0.169		3.92(7)	61.6%
	1.478	0.429	0.736		

C1P15 (*)

Grn 3.05	0.575	0.179		4.00	62.8%
	1.460(5)	0.455	0.762(5)		

(*) no DOCA on final pellets - Tom took

1/19 RUNS IN FURNACE AS %. all RAMPS 10°C/min

HOOD	2 HRS		: SINTER TIME
	FURNACE 1	FURNACE 2	
	(12) (13)	(14) (15)	

1/19 A.M. ⇓

4:10 P.M. 4:25 P.M.

∴ to temp (975 C)

6:1[illegible] P.M.

RAMP down to 600C SOAK

1/20 1:[illegible]9 PM.

RAMP down to RT

CHECK 2:22 (270C).

Pellet thickness experiment DD mill pwdr 3775/26,000

DT2.0

2.04	0.575	0.119		4.03	63.3 %
2.01	1.460(5)	0.302	0.506		
	0.507	0.100		6.09	
	1.288	0.254	0.33		95.6

DT 1.5

1.54	0.575	0.090		4.01	62.95 %
1.51	1.460(5	0.229	0.384		
	0.509	0.075		6.04	94.8
	1.293	0.190(5)	0.250		

DT 1.0 K

1.09	0.575	0.065		3.95	62.0 %
	1.460(5)	0.165	0.276		

Cutting Calculations for C1P12, 13

0.~~5~~93
0.06
0.~~0~~93/3 = 0.0~~3~~31 3 blade thickness + 0.0[illegible]

C1P12 (0.025) 5 = 0.125
(↓) 6 = 0.150 ← O.K. from micrometer

use 2 cuts { no 'parallelng'

0.~~0~~50
0.040
0.11/3 = 0.037 + 0.015 = 0.0517
to from edge

1 cut MADE, BUT PELLET HAS CRACK

(0.025) 6 = 0.150
0.040
0.11/3 = 0.037 + 0.015 = 0.052

1/21

DT 1.751 IN furnace/no green data (5°C RAMP to try to eliminate SINTER-CRACKING)

DT 1.75(2) never RUN
1.88 0.575 0.111 3.98 62.5
1.4605 0.282 0.472

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Stereopycnometer 1/27/88 {25/26 supply, miller repairs}
28
29 2/01

See sheets

Data Points (Multiples)

	D_M		D_P	
"83"	82.95	DRC, DDP12	95.8	NOTE pack: 97.3 − 95.8
"86"	86.4	JP2b2, C1P3, C1fp2	92.2	← Δ 86.4 − 89.3 = Δ 3%
"89"	89.2	C1fp1, C1p4, C1p7	89.56	
"91"	91.3	C1p1, C1p5, C1p8	91.9	

Single Point trends ⊗

87.5	87.5	JP1	83	NOT clear, seems to ~~[illegible]~~ be closed
77		C2P2	95.4	DEFINITELY stuck open !
"93"	93	DDP13	86.6	indicates closure

⊗ small volumes yield low D_P values for closed porosity.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

3500/26,000

2/02 Sintering: Porosity Inquiry. C1 & C2 @ 975

{ 10°/min Ramp from RT, 2 hour soak, 10°/⇒ to RT no O_2 equilibration. In order from left to right in oven,

C1P16

3.03	0.575	0.178		4.00	62.8	green, polished
	1.460(5)	0.452	0.757			
3.00	0.508	0.159		5.92	92.9	
	1.293	0.386	0.507			

C1P17

3.26	0.575	0.191		4.01	62.9(5)	green
	1.460(5)	0.485	0.812(5)			
3.22	0.508	0.164		5.94	92.8	
	1.290	0.4166	0.544(5)			

C2P6

	0.575	0.194		3.82(6)	60.0	green
3.16	1.460(5)	0.493	0.826			
3.11	0.497	0.168		6.12	96.1	
	1.262	0.406	0.507(8)			

C2P7 chip ⊛

	0.575	0.199		3.79	59.5 ⊛	green, polished
3.21	1.460(5)	0.505(5)	0.847			
3.16	0.497	0.164		6.065	95.2	
	1.262	0.4166	0.521			

C2P7 good { dense, but exterior cracking due to oxygen penetration. Will quick cool by opening furnace. Quench!

ITEM						
C2-8	3.08	0.523	0.191		3.82	59.97 ⇒ ~60% comparable to previous see 86.8
		1.455	0.485	0.8016		
	3.06(5)	0.529	0.156		5.53	
		1.328	0.399	0.553		
C1-18	3.07	0.578	0.178		4.01	62.95 ~ 63% comparable to previous 94.8
		1.468	0.452	0.765		
	3.03	0.497	0.158		6.04	
		1.262	0.401	0.5016		
2/12 HP-4	green	5,000/27,000				
	13.98	0.947	~0.301		4.02(5)	63.2
		2.405	0.764(5)	3.473		

Cutting C2-8 dry
1st 0.50

2/15

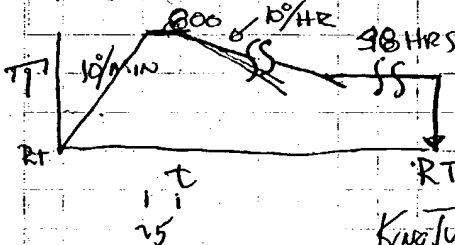
Boat Spots → positioning I.D.

(1 2 3 4 5 6 7) (45MIN)

1. } large grain material { C1 3/4
2. }

Heat Treatment

3. } C2 std. w/ surface cracking (outer)
4. } INNER SURFACES should be O.K.



5. Good C2 top slice - NO surface cracking
6. middle slice of 3/4 (w/ chip)
7. bottom slice of C2-5 above.

King IV →

START: 4:50 PM 2/12 → 6:15 start ramp down / 7:00 20HRS to soak point ~ 30HRS

~~20HRS~~ -3:00

QUENCH 2:50 2/15

3500/26750

C2-9	3.09	0.577	0.193		3.76	59.0%
		1.460(5)	0.490	0.821		
	3.06	0.510	0.168			85.5
		1.295	0.427	0.562		
C2-10	3.06	0.575	0.191		3.77	59.2%
		1.460(5)	0.485	0.812(5)		
	3.0(5)	0.501	0.164		5.77	~~90.6~~
	3	1.272(5)	0.417	~~1.29~~		89.6
				0.530		

Furnace O2 purge > 1 HR @ 29(32)°C 12:10 PM, .945/10 =
94.5 mins/60min/HR = 1.575 Hrs OR 1 hr 34.5 mins (1:45 START SINTER)
1:45 - 2:15 (1/2 hr sinter) w/ quench.

C2-11	3.02	0.575	0.188		3.775	59.3% O.K.
		1.460(5)	0.477(5)	0.80		
	2.98	0.505	0.159		5.71	89.64 ~90
		1.283	0.404	0.522		

The above understood and witnessed by _____ Date _____ and by _____ Date _____

D≤4μm D≤6μm D≤6-7 D≤9

MICROMERITICS CAPA-500
PARTICLE ANALYZER

DATE 2/18
SAMPLE C2-III.01
SOLVENT 150
D> 0.83

• CONDITIONS

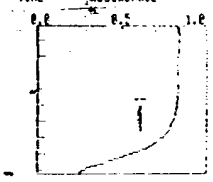
SOLV.VISC 2.16(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.37(G/CC)
D(MAX) 16.0 (MI...
D(MIN) 1.00(...
D(DIS) 1.00(MI...

SPEED 500. (RPM)

• TIME 0 H 4 MIN 19 SEC

• DATE

TIME ABSORBANCE

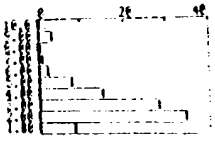


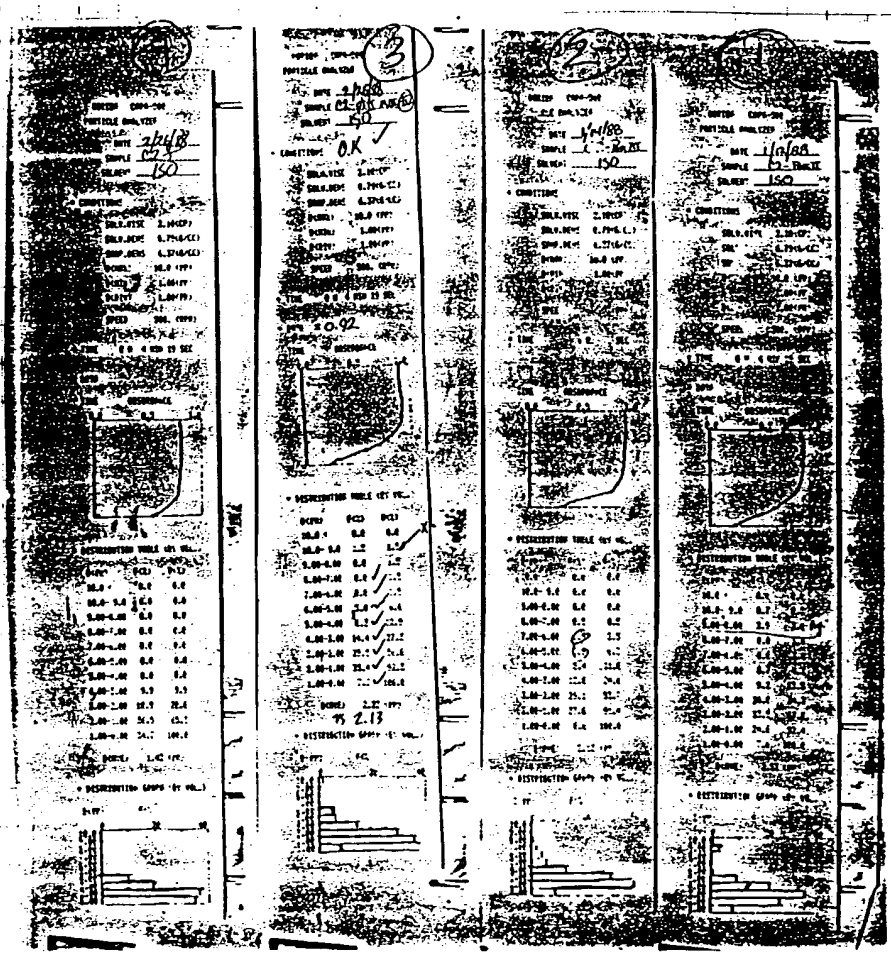
• DISTRIBUTION TABLE (BY VOL.)

D(MI)	F(%)	R(%)
16.0 (	0.0	0.0
16.0- 9.0	0.0	0.0
9.00-8.00	2.9	2.9
8.00-7.00	0.3	3.2
7.00-6.00	0.7	3.9
6.00-5.00	1.8	5.7
5.00-4.00	7.8	13.5
4.00-3.00	15.1	28.6
3.00-2.00	28.2	56.8
2.00-1.00	34.7	91.5
1.00-0.00	8.5	100.0

D(AVE): 2.24 (MI...

• DISTRIBUTION GRAPH (BY VOL.)





C2 PSDs

1) C2 MILL PASS II
2) C2 ↓ PASS III of 1/2 of ①
③ C2 MILL PASS IV ⊗ of other 1/2 of ①
④ fines from ①, ②, ③ III
⊗ 3rd milling pass ineffective due to clogged bag & pwdr. charged channels.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

36 $SrTiO_3$ 3/4 Synthesis (see Book III page 97 for work-up)
: No comp → 69's, 39's

Prep:

tare 206.15

$SrCO_3$	50.00 g	desired
	256.15	
	256.15/6	actual wght
	0.0	Δ
TiO_2	27.062	desired
	283.212	desired
	283.22	actual wgh
	+0.01	Δ
	+0.01	scale replace
	~0.0	Δ net

1 hr + mixing

TRANSFER

tare 89.20

166.23	final wght
77.03+	
77.062	expected
-0.03 g's	Δ 0.04

185.56 total prelim. wght w/ top
19.33 g top

theoretical expected 151.36 w/out top 150.97
19.33
170.69 w/ top

3/5

14.87 + 62.16 = 77.03 ~ correct

Ramp @ 700C/HR to 1450C ⇒ to temp ~ 3:25

0.39/150.97 (0.258% loss)

GROUND yield ⇒ 61.39/62.16 ⇒ 98.8%
~1% grinding loss

CLEAN X-RAY. Milled 1 HOUR.

SYN PROJECT COMPLETE 3/5

The above understood and witnessed by _____ Date _____ and by _____ Date _____

3/4/88

C2 pellets C2p12-15 batch I mill III
C2p16-17 2 IV

3700/27000

C2p12 3/21 → page 44	~~3.075~~ (3.08)	0.572 1.453	0.191 0.485	0.804	3.823	60.0
C2p13 3/21 → page 44	3.02 (3.02)	0.573 1.455	0.188 0.477(5)	0.794	3.803(5)	59.7
C2p14 page 47	3.11	0.574 1.458	0.192(3) 0.488	0.815	3.82	59.9
C2p15 page 47	3.11	0.574(5) 1.459	0.192(3) 0.488	0.816		59.8
C2p16	3.25	0.573 1.455	0.202 0.513	0.853		59.8
C2p17 sue chopped	3.22 add 0.02 (3.24)	0.573 1.455	0.202 0.513	0.853		59.6+

The above understood and witnessed by _____ Date _____ and by _____ Date _____

$SrTiO_3$ GB Doping

10 g $SrTiO_3$ w/ 2 wt% $B_{12}O_3$ added

Sp. g - 8.8 m.p. 820°C

10 g + 0.2 g $B_{12}O_3$ => 10.2

0.2 g A_2O 7.14 g/cc Decomposes above 300C

$AgNO_3$ >> mp 212C bp => decomp 169.8749 mw

~~4.328~~ g/cc

$0.2\,g\,Ag_2O \times \frac{169.8749\ g\ AgNO_3/M}{231.7394\ g\ Ag_2O/M} \rightarrow 0.733, 1.364$

$0.2\,g\,Ag_2O \times$ ~~$\frac{169.8749\ g\ AgNO_3/cc}{231.7394\ g\ Ag_2O/cc}$~~ $= 0.1466 \approx 0.15\,g$ OK

X2 = 0.29

$0.2\,g\,AgNO_3 \times \frac{169.874\,g}{}$

$2\,AgNO_3 \xrightarrow{\Delta} Ag_2O + 2NO_2 + O$

~~$0.2\,g\,Ag_2O \times \frac{231.7394\ g\ Ag_2O/M}{169.8749\ g\ AgNO_3/M} = 0.27\,g\ AgNO_3$~~

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Free

I-27500 Doped $SrTiO_3$ pellets

$SrTiO_3$ basis 3/5

STA-1	3.10	0.581	0.196		STA-3 deformed, save pieces,	3.64	75.7
		1.476	0.498	0.852			
	3.0	0.525	0.178			4.75	98.8
		1.334	0.452	0.632			
STA-2	3.25	0.581	0.208			3.65	75.9
		1.476	0.521	0.891			
	3.14	0.525	0.185			4.78	99.4 ← polish 1
		1.334	0.470	0.657			
I-24 STB-1	3.03	0.587	0.191			3.58	74.4
		1.491	0.485	0.847			
2.88 ~~<2.92~~ (dup)		0.590	0.179			4.49	92.9
		1.370	~~0.492~~ 0.437	0.~~64~~ 74)			
STB-2	3.17	0.586	0.197			3.64~~6~~	75.9
		1.488	0.500	0.869(5)			
3.03 ~~<3.21~~			0.179			4.52	~~94.~~
		1.370	0.455	0.671			
I-27500 STB-3	3.77	0.583	0.237			3.66	76.1
		1.481	0.60	1.03			
3.61 ~~<3.74"~~ deformed		0.534	~0.216			4.55	99.6 ← polish 1
		1.366	0.549	0.793			
ST-D1	3.62	0.585	~0.235 varies	1.03(5)		3.50	72.8
		1.486	0.597				
3.60 ~~<3.67"~~		0.527	0.210			4.77	99.2+ ← polish 1
		1.339	0.533	0.7505			

Comments - green D's fairly consistent, even w/ pressure variation

The above understood and witnessed by _____ Date _____ and by _____ Date _____

37/27000

DD-X	2.88	0.573	0.169		4.04	63.4%
		1.455	0.429	0.713		
	2.83	0.508	0.144			93
		1.290	0.366	0.478		
DD-Y	2.99	0.575	0.174		4.04	63.4%
		1.460(5)	0.442	0.740(5)		
	2.93	0.509	0.149			92.4
		1.293	0.379	0.498		

10°C/min ramp. in new Al_2O_3 crucible on fresh DD powder.
975C for 2 hours & quench. 20min O_2 purge.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

HORIBA CAPA-500
PARTICLE ANALYZER
DATE Bi_2O_3
SAMPLE 2/24
SOLVENT ISO

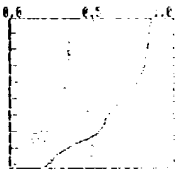
• CONDITIONS

SOLV.VISC	2.79(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.88(G/CC)
D(MAX)	10.0 (FM)
D(MIN)	1.00(FM)
D(DIV)	1.00(FM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 0 SEC

• DATA

TIME ABSORBANCE

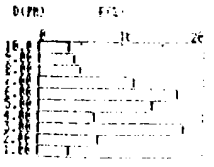


• DISTRIBUTION TABLE (BY VO...

D(FM)	F(%)	R(%)
10.0 <	10.6	10.6
10.0- 9.0	3.5	13.9
9.00-8.00	4.2	17.2
8.00-7.00	4.8	22.0
7.00-6.00	11.8	34.1
6.00-5.00	16.8	50.9
5.00-4.00	13.6	64.7
4.00-3.00	6.8	71.5
3.00-2.00	17.4	88.9
2.00-1.00	7.2	96.1
1.00-0.00	3.7	100.0

D(AVE) 5.8 (FM)

• DISTRIBUTION GRAPH (BY VO...



HORIBA CAPA-500
PARTICLE ANALYZER
DATE Bi_2O_3
SAMPLE 2/24
SOLVENT ISO

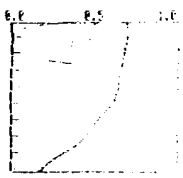
• CONDITIONS $V = 0.69$

SOLV.VISC	2.79(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.88(G/CC)
D(MAX)	10.0 (FM)
D(MIN)	1.00(FM)
D(DIV)	1.00(FM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 0 SEC

• DATA

TIME ABSORBANCE

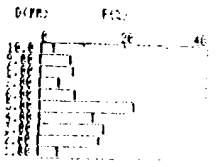


• DISTRIBUTION TABLE (BY VO...

D(FM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.9	2.9
9.00-8.00	7.1	10.0
8.00-7.00	7.6	17.7
7.00-6.00	4.4	22.1
6.00-5.00	8.3	30.4
5.00-4.00	22.4	52.8
4.00-3.00	12.8	65.6
3.00-2.00	15.5	81.1
2.00-1.00	14.5	95.6
1.00-0.00	4.5	100.0

D(AVE) 4.12 (FM)

• DISTRIBUTION GRAPH (BY VO...



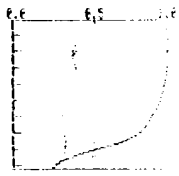
HORIBA CAPA-500
PARTICLE ANALYZER
DATE 2/24/88
SAMPLE C_2F_{111} PASS III
SOLVENT ISO
in Sector milling
• CONDITIONS clean motor

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (FM)
D(MIN)	1.00(FM)
D(DIV)	1.00(FM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

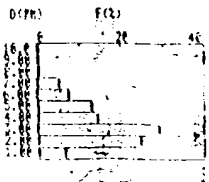


• DISTRIBUTION TABLE (BY VO...

D(FM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	5.1	5.1
6.00-5.00	7.6	12.7
5.00-4.00	12.8	25.5
4.00-3.00	14.3	39.8
3.00-2.00	29.7	69.6
2.00-1.00	24.7	93.3
1.00-0.00	6.7	100.0

D(AVE) 2.65 (FM)

• DISTRIBUTION GRAPH (BY VO...



The above understood and witnessed by _____ Date _____ and by _____ Date _____

3/15 DC batch II $SrTiO_3$

jar tare 206.11 (206.
256.06(7)
49.96 g loss 0.05 (0.1%) desired 50g unstable

27.062
283.122 target
283.13 actual ✓
27.07 +0.008
77.02 total

88.34(5) Pt cruc tare
77.02 total above
165.36(7) expected comb. wgt
165.34
.03 g error max. ✓✓ OK. (0.04% error)

~184.54 (19.20 tare) ✓✓ expect ~150.0 w/out top

~~10.88~~ 150.15 after cooling!

3/16 41.20 < 100 ~~mesh~~ = 59.85 g

ST-D2 (2.9μm) MII	3750/25,000 3.04	0.583 1.481	0.206 0.523	0.900		3.38	70.3 %
	3.017	0.514 1.306	0.181 0.460	0.616		4.90	1.02 %

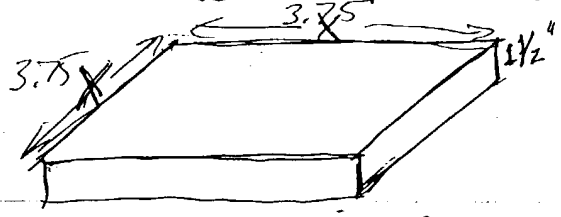
The above understood and witnessed by _____ Date _____ and by _____ Date _____

7070 GLASS count

1½"

7070 density - 2.13 g/cc ⇒ $\frac{kg}{100g} \times = \frac{0.00571\ lbs}{cc}$ $3.73 \times 10^{-1} kg$ / lb

$0.00571 \frac{lbs}{cc} \times \frac{16.387\ cc}{IN^3} = 0.0936 \frac{lbs}{IN^3}$ troy conv.



$4.08 \times 4.08 \times 1.5 = 21.32\ IN^3$

$21.32 \times 0.08 \frac{lbs}{IN^3} = 1.99$ lbs

24.9696

$1.5 X^2 (0.08 \frac{lbs}{IN^3}) = 2$

$1.5 (0.08\ lbs/IN^3) X^2 = 2\ lbs$ 4.08^2

$0.12 X^2 = 2\ lbs$

$X^2 \simeq 16.66$

$X = 4.08$

check density conversion: $2.13 \frac{kg}{cc} \times \frac{lb}{0.435\ kg} =$

OK $\frac{0.00213\ kg}{cc} \times \frac{1\ lb}{0.435\ kg} = 0.0048965 \frac{lb}{cc}$

$1\ lb = 4.535 \times 10^{-1}\ kg$
$lb = 0.435\ kg$

$0.0048965 \frac{lb}{cc} \times \frac{16.387\ cc}{IN^3} = 0.08$

4 X 4 X 1.5 OR 5 X 5 X 1

@ 1" thick $0.08 X^2 = 2$

$X^2 = 25$

$X = 5$

The above understood and witnessed by _____ Date _____ and by _____ Date _____

3/21 1ST pellet 700C for 12 hrs ⇒ START @ 3 to RAMP @ 10°/MIN to SOAK for 16-17 hrs.

C2P12 for green data on all pellets ... pg 37

C2P13 2nd pellet 750C Don't to Run concurrently

Peter,

Since we didn't get to discuss this experiment in more detail, here is what needs to happen.

5 pellets — C2

		overnight	Init.
1st)	700° C	~12 hr O_2	3.08
2nd)	750° C	~12 hr O_2	3.02
3rd)	800° C	" "	
4th)	850° C	" "	

After these intermediate temperature anneal, weigh and measure each pellet. If no sintering, or at least a negligible amount, has occured, then refire each sample for 12 hrs again at the same intermediate temperature and then sinter each pellet for 2 hrs at (950)°C. Ramp from the intermediate T to (950°C) fast (~20°C/mn).

Also sinter the 5th pellet at 950°C for 2 hrs, this is the control pellet. Thanks and have a good week.

Duane

} 965C used

3/24-9:00 AM, w/17 HRS

C2P12 3.06 (Δ-0.02) 0.572 0.191 no sintering, but 0.65% weight loss
3.01 0.50 0.163 5.65 88.7
1.27 0.419 0.533 CRACKING = closing

3/23: Temp RAISED to 950 @ 9:00 AM
> to temp @ 9:15 am

C2P13 3.01 (Δ-0.005) (Δ-0.01) 0.572 0.187 no sintering, but 0.60% weight loss
2.97 0.504 0.163 5.57 87.4
1.280 0.414 0.533 NO CRACKING - "open"

The above understood and witnessed by _____ Date _____ and by _____ Date _____

3/21 $SrTiO_3$ DRC-batch2 ⇒ fine cut after 3rd milling ⇒ 11g yield
+ 50g medium.
61g / 72 orig.
~ 85% yield

after cleaning ~ 2g loss to machine
3g loss to blow-out

approx. expectations { 18% fines 82% medium

fines ⇒ 1.34 μm ave d
medium III ⇒ ≥ 2.2 μm RANGE (2.2-2.8)

STD 1f-1	0.570	0.212		3.09	64.2 % {versus 72 on 3um pucks
2.74	1.448	0.538(5)	0.887		

In furnace ~ 3:00 p.m., tripped off @ 975 2X, cooled to 1400C, then to 1600C.
Temp recovered/reset to 1650 @ ~4:30 p.m..

3/22 RAN all nite! 24 HRS @ 3:00 p.m. tuesday, 42 HRS. @ 9:00AM WEDS.

2.70	0.487	0.178	4.97	1.033 % same as dd's
	1.237	0.452	0.543	

Peter:

✓ started • Cut, section and polish Cu-Bi slab (Hot Plate) start/finish 21/22

• Try firing one pellet of $SrTiO_3$ @ 1350C overnight

• Try slip casting a pellet of $SrTiO_3$

→ fines Jet mill $SrTiO_3$ powder down to ~1μm. This will require "tuning" the jet mill. Then fire 1650C overnight USED fines, fire 21/22

✓ • Make master batch of $SrTiO_3$?

tare 202.33 *approx. due to instability of spans dose: sometimes stable
50.00
252.33 target
act wght 252.33 * wt
Δ 0.0

27.062
279.392
act weight 279.4
~Δ 0.01

TRANSTEK. 165.22 act wght
tare 88.17
77.05 wght mix
77.07 expected
~0.03% loss

POST/16HR 150.02
+ 14.87 g ~expected loss
164.89
15.2g actual loss

The above understood and witnessed by Date and by Date

write-up 3/23, purde 3/24 MONN

46 3/22

IBM Technical Notebook

C3-Synthesis (Reference)

Synthesis

$BaCO_3$

tare ⇒ 277.72 to zero

weight: 7

3/23 398.24-6 total

120.52-54 (-0.02%)

CuO

tare ⇒ 0.89 ⇒ zeroed

weight: 72.58 (7/9) 3/23

transferred quant

Y_2O_3 tare · 0.85 ⇒ z'd 3/23

weight 34.35

transferred quant, +0.03%

total expected ⇒ 227.46

14 C2 Batch → $YBa_2Cu_3O_7$ 200g

from C1 batch calc. (99.9% Batch III) 72 Batch II)
wet feac. Ready Mult. Found

Y_2O_3 ⇒ 17.1535 ⇒ 17.1907 ⇒ x2 (34.34)

BaO_X ⇒ 46.3934 100 ⇒ x2 93.1868

$BaCO_3$ conversion: 93.1868 × 197.35/153.34 = 119.932(5) ÷ 0.99 ⇒ 121.14(9)

CuO ⇒ 36.25(5) ⇒ 36.2895(9) ⇒ x2 (72.57(9))

O.K. weighing in Ba rich by analysis, so dry not not correct ⇒ 119.93 (120.54)
Apply ½.

$BaCO_3$

tare: 279.67
120.59
100.21 won't need, let all tare

reads: 120.57(46) ooo 4/5

CuO

tare: 0.89/7
reads 72.58 transferred quant. tare to zero of paper

Y_2O_3 transferred quant
34.34/5 paper weight 0.1 after checked due to static from dry box, after dump/dump several 0.00. Back OK after careful glove use (not more than 0.3% error)

Expected total weight 227.46 g day

BaO - 5.72 g/m $BaCO_3$ - 9.93 Y_2O_3 - 5.01 CuO - 65-6.49

∴ If bumping occurs or solution loss, $BaCO_3$ should preferentially be lost & not ~~not~~ uniformly suspended.

Overnite @ 70C in 30" vacuum after "bump-free" isopropanol mixing

3/24 #1 cruy 230.56 − 116.57 = 113.99 + #2 230.41 − 117.17 = 113.24 ⇒ 227.23 · 0.1% loss or 99.9% Recovery

Prior to removing from bkr after overnite, cake broken up and "pudrized", then let cool under vacuum to remove any soln. resid.

In furnace @ 12:30 500C/HR RAMPS, 955 RXN T in flowing oxygen

POST 3/25 219.22 − 102.65 = 101.79 + 218.34 − 101.17 = 100.34 ⇒ total 203.82 − 202.13 = 0.83% loss

218.36

318.71

The above understood and witnessed by _____ Date _____ and by _____ Date _____

3/23 from pg 44 TREATMENT INQUIRY

C2P14 & C2P15 ⇒ original green δ info on pg 37; both 3.11 then & now

C2P14: (800°C pretreat), purge - 2120 p.m. 59.9 n.d. ≈ 18 HRS

3.09	0.573	0.191		3.83	60.1	no appreciable sintering
	1.455	0.485	0.806			
3.07	0.522	0.171		5.125	80.5	apparently sintering appreciable
	1.326	0.434	0.599			

C2P15: (850°C pretreat) purge as above - 59.8 n.d. ≈ 18 HRS

3.09	0.565	0.187		4.02	63.1	slight amount of sintering
	1.435	0.475	0.768			
3.08	0.531	0.174		4.87	76.5%	initial sintering "appreciable
	1.349	0.442	0.632			

C2P18 (CONTROL) 37/27500

2.92	0.574	0.180		3.83	60.1%	O.K.
	1.458	0.457	0.763			
2.86	0.501	0.152		5.82	91.4	⇐
3/28	1.273	0.386	0.491			

Post 48 hr. (2nd 24) - C3 batch

pre ⇒ 318.71
post ⇒ 316.88
(-) 1.83
202.13
200.30
199.23 initial grd. yield

STD-1f grain size slightly larger – interior fairly uniform ~25 μm ave by occasional 50 μm grain

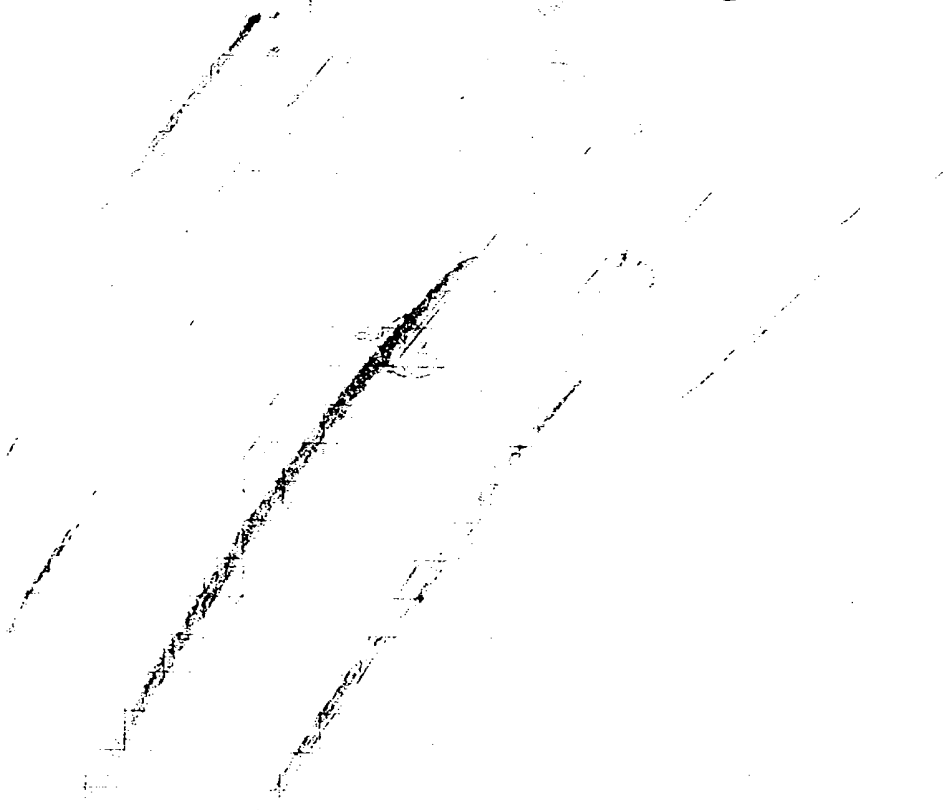
~~Refeed~~ Re-sintered overnite to check for additional growth. Further polishing of 40 hr sample slice yields numerous 40-50 μm GRAINS! Growth seems probable!

3/24

Summary from 45

Green	2.721	1.498	0.538(5)	0.887	3.09	64.2
40 HRS @ 1645C	2.70	1.237	0.452	0.543	4.97	100.33

slice back up to 1650 @ 5:00 PM (1 hr earlier due to control couple failure)
63 HRS SHOT OFF @ 1:45 PM 3/24 (42 1/2 HR)



The above understood and witnessed by _____ Date _____ and _____ Date _____

3/24 from pg 45

after additional 12 hr RXN time: 150.02 − 149.81 = 0.21 g ; Assume constant now

total loss: 165.22 initial Δ

150.02	16 HR	−15.20	98.6 % Reacted
149.81	28 HR	− .21	1.4 % ? CASE FOR MINOR porosity?
		15.41	

> 61 gs recovered after mortar grinding.
> 2 HRS on shaker mill w/ 5mm balls.
> 60.4 g shaker yield
> 78.8 g MI JET YIELD

PSD
2.91 μm ave. ∧ Slightly finer jet, but not much better D.size.

Slip-cast calculations: die 0.9" ∅ ⇒ 2.286 cm ; 0.3" (0.762 cm) desired green thickness

$\frac{\pi \cdot (2.286)^2}{4}(0.762) = 3.1275$ cc (4.81 g/cc) = 15 g Si_3N_4 (0.6) = 9.0 g

(0.6 ↑ approx density)

The above understood and witnessed by _____ Date _____ and by _____ Date _____

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/21/88
SAMPLE SrTiO3-DRC2
SOLVENT IS0
MIII

• CONDITIONS

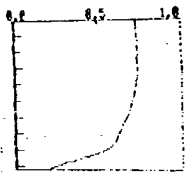
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



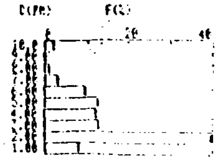
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.0	1.0
9.00-8.00	0.6	1.6
8.00-7.00	1.6	2.6
7.00-6.00	2.9	5.5
6.00-5.00	9.5	15.0
5.00-4.00	12.5	27.[illegible]
4.00-3.00	12.1	39.8
3.00-2.00	12.6	52.5
2.00-1.00	39.6	92.[illegible]
1.00-0.00	7.9	100.0

D(AVE) 2.20 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE SrTiO3
SOLVENT
MII

• CONDITIONS

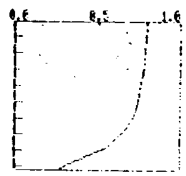
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



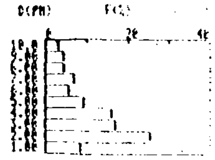
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	3.1	3.1
9.00-8.00	4.2	7.3
8.00-7.00	4.5	11.9
7.00-6.00	6.9	18.8
6.00-5.00	5.4	24.2
5.00-4.00	9.2	33.4
4.00-3.00	16.6	49.4
3.00-2.00	17.0	66.[illegible]
2.00-1.00	25.2	91.6
1.00-0.00	8.4	100.0

D(AVE) 2.96 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



DRS

HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE SiTiO3
SOLVENT
MIII

• CONDITIONS

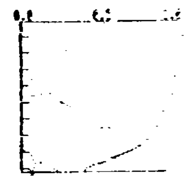
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



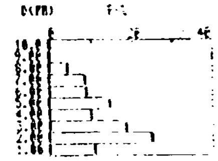
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	[illegible]	[illegible]
8.00-7.00	4.[illegible]	4.[illegible]
7.00-6.00	8.4	12.[illegible]
6.00-5.00	8.7	21.6
5.00-4.00	14.4	36.[illegible]
4.00-3.00	9.5	45.5
3.00-2.00	18.4	64.[illegible]
2.00-1.00	24.5	89.[illegible]
1.00-0.00	10.7	100.0

D(AVE) 2.70 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/21/88
SAMPLE SrTiO3-DRC2
SOLVENT IS0
MIII-5

• CONDITIONS

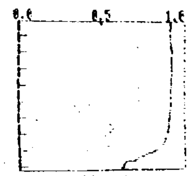
SOLV.VISC 2.10(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 4.81(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 6 MIN 0 SEC

• DATA

TIME ABSORBANCE



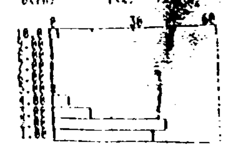
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.9	2.9
9.00-8.00	0.0	2.9
8.00-7.00	0.6	2.9
7.00-6.00	0.0	2.9
6.00-5.00	0.0	2.9
5.00-4.00	0.0	3.0
4.00-3.00	5.9	9.6
3.00-2.00	13.4	23.0
2.00-1.00	41.0	64.[illegible]
1.00-0.00	35.9	100.0

D(AVE) 1.34 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



3/29

10 g $SrCO_3$ ⇒ 0.06774 moles
5.4112 g TiO_2
15.4112 g total
15.08 after mixing Recovery
.33

9/25/93 Note
Density calculations here were done using 5.116 lit. which is now known to be in error. It is 5.116 not 4.82

Dave's unreacted $SrCO_3/TiO_2$ } new batch $SrTiO_3$ ⊗

2.975	0.584	0.242				
2.92	0.458	0.190		4.745 !	98.6 !	not too good
	1:16	0.483	0.510			
⊗ 2.91	0.585	0.194				
2.90	0.588	0.173		4.785	99.5 !	not too good
	1.326	0.439	0.606			

The above understood and witnessed by _____ Date _____ and by _____ Date _____

SrTiO3 grain growth 1470C

pellet 2 of fewes

2.22	0.572	0.169		3.12	64.9%	~same as before
	1.453	0.429	0.711			
2.19	0.487	0.144		4.98	~~100.5%~~	= 103.5
	1.237	0.366	0.440			
2.71	0.580	0.176		3.56	74.0%	
Dianes xcess	1.473	0.447	0.762			
TiO_2						
estimate on ½	0.518	(0.262) 0.160		4.86	→ 101.0	101.—
eb.35	1.32	0.406	0.556			
(2.70)	~~1.31~~	~~0.404~~				
~ O.K. by wght.						

1

The above understood Date and Date

$SrTiO_3$ doping

½ mole % TiO_2

		X2	M.W.	M.P.	
0.0676	0.1352 5g batches		79.90	~~0.0676~~	
0.125 excess	0.25		147.63		
~~0.125~~					
STOIC	as nitrate		101.96		
0.086	SAME				
0.154	0.308		181.88	3.357	690C
			183.5182		

		moles	
13.531	TiO_2	0.16935	0.00084675 = ½ mole %
25.00	$SrCO_3$	↓	
38.531			

0.00084675 moles (79.90) → -0.06766 + 13.531 = 13.46

0.00084675 (147.63) → -0.125 + 25. = 24.875

Summary of additions, quantities

	excess	SUB 1	SUB 2	excess 2
TiO_2 (g)	13.5986 13.6662	13.445 *	13.531 *	13.~~55~~
X2 $SrCO_3$	25.00 ↓	25.00 ↓	24.~~8~~46	25.~~2~~5
X2 ~~X2~~ Al_2O_3	—	0.086*	—	—
V_2O_5	—	—	0.154*	—
			0.16	

* these quants are X2 since there are 2 moles of Al & V in Al_2O_3 & V_2O_5

Correction: $\frac{.16}{147.63}$ = 0.00108 moles $SrCO_3$

$\frac{0.00108 \text{ moles}}{0.00169}$ = {64% with loss due to decanting approx 70% stoic or 30% off addition excess V_2O_5}

The above understood and witnessed by _____ Date _____ and by _____ Date _____

IBM Technical Notebook

Sub2 V_2O_5 1 mol %, stoichiometric / not excess (see pg 53)

25.0 g $SrCO_3$ weighed & transferred to beaker
0.16 g removed
0.16 g V_2O_5 added (~20cc saturated in hot water, decanted)

MISTAKE, now uncorrectable. Should have been:

0.00084675 (2) = 0.0016935 g $SrCO_3$ removed ∴

0.0016935 (147.63) = 0.25 g however, decanting over residual pads reduced actual V_2O_5 addition, AND though NONSTOICHIOMETRIC (slightly) will use to see what happens.

38.62 yield after overnite vac @ ~90C
38.53 initial
0.51 loss in mixing 1.3 %

88.88 tare (zeroed)
38.08 466 extra due to final beaker scrape ✓

In furnace to temp by 12:00, 4/6/88 16 HRS 8 A.M 4/7

'Severe' sintering, dark black appearance of pads body.

126.96
117.68
~.05 g spillage
117.73

25g $SrCO_3$ × $\frac{\sim 103.62}{147.63}$ ≃ 17.55

~7.40 g expected loss

126.96
−117.68
9.28 ! some bound water?

26.18 g ground yield

SEE 57 & 58 FOR SINTERED

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Sub 1 Al_2O_3 → 1 mol% added as nitrate

0.0008475 (2) = 0.0016935 moles // $Al(NO_3)_3 \cdot 9H_2O$ 375.14

½ mol% 1 mole % Al 1:1 so use 0.0016935 moles

0.0016935 moles Al nitrate $\left(\frac{375.14\,g}{mole}\right)$ = 0.6353 g

(303172)

So remove 0.0016935 moles TiO_2 ∴ 0.0016935 (79.9) = 0.135 g TiO_2

13.531
− 0.135
13.396 g TiO_2, 0.6353 g Al nitrate in soln

38.29 mix yield
39.03 theoretical
0.74 mix loss 1.9%

$0.6353\,g\left(\frac{101.9612}{375.14}\right) = 0.173$ − Δ0.16

39.03
− 0.96
38.57
38.27 ⇒ 0.3+ mix loss ✓

89.55 tare (fired)
38.27 NOTE: nitrate decomposes in hot water. Must explain some of loss

In furnace to temp by 12:00 p.m., 4/6/88 ⇒ 16 hrs 8 AM 4/7

Little sintering of pwdr., light $SrTiO_3$ color, mottled.

118.54
127.82
− 9.28 g ! same as SUB 2 ! Good proving the same, even though exact loss is coincidence.

yield → 27 g

see 57 | 58 for SINTERED DATA

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Those Diagrams (arrows) for $SrO \cdot TiO_2$ system are absorbed ones

Two Oxides 119 Figs. 296–301

$SrO-SiO_2$

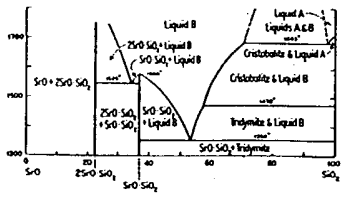


FIG. 296.—System $SrO-SiO_2$.

P. Rakola, *Am. J. Sci.*, 5th Ser., 4, 336 (1922); modified by J. W. Greig, *ibid.*, 5th Ser., 13, 19 (1927); see also F. C. Kracek, *J. Am. Chem. Soc.*, 52 [4] 1440 (1930).

$SrO-TiO_2$

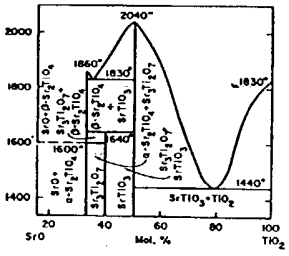


FIG. 297.—System $SrO-TiO_2$.

Miroslawa Dryś and Włodzimierz Trzebiatowski, *Roczniki Chem.*, 31, 492 (1957).

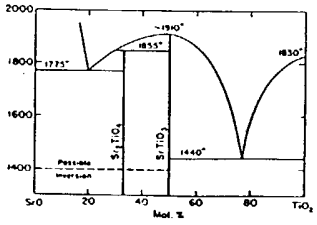


FIG. 298.—System $SrO-TiO_2$; tentative.

Rustum Roy; private communication, 1957.

$ZnO-Al_2O_3$

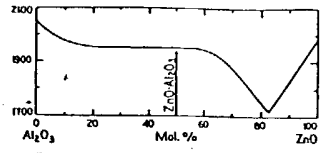


FIG. 299.—Liquidus curve of system $ZnO-Al_2O_3$.

E. N. Bunting, *Bur. Standards J. Research*, 8 [2] 280 (1932); R. P. 413.

$ZnO-B_2O_3$

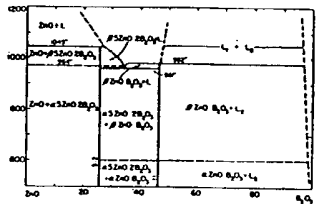


FIG. 300.—System $ZnO-B_2O_3$.

D. E. Harrison and F. A. Hummel, *J. Electrochem. Soc.*, 103 [9] 496 (1956); see also, "Structure of Zinc Metaborate, $Zn_3O(BO_2)_4$," P. Smith, S. Garcia-Blanco, and L. Revoir, *Anales Real Soc. Espan. Fis. Quim.* (Madrid) *Ser. A* (Nov.–Dec.), 263–268 (1961).

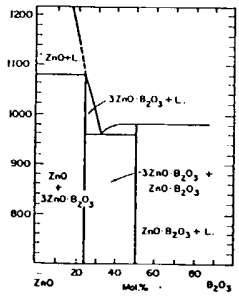


FIG. 301.—System $ZnO-B_2O_3$.

Yu. S. Leonov, *Zhur. Neorg. Khim.*, 3, 1246 (1958).

Two Oxides 93 Figs. 2334–2336

$SrO-TiO_2$

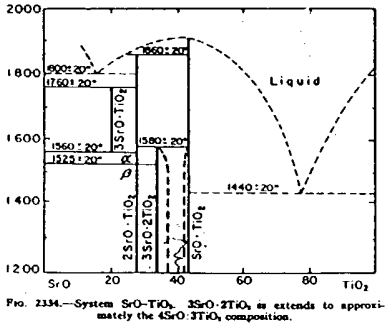


FIG. 2334.—System $SrO-TiO_2$. $3SrO \cdot 2TiO_2$ as extends to approximately the $4SrO \cdot 3TiO_2$ composition.

Antonio Cocco and Franco Massazza, *Ann. Chim. (Rome)*, 53, 892 (1963).

$SrO-ZrO_2$

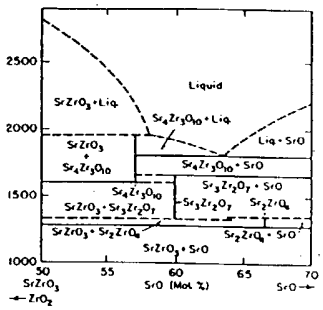


FIG. 2335.—System $SrO-SrZrO_3$.

Gilbert Tilloca and Monique Perez y Jorba, *Rev. Hautes Temp. Refractaires*, 1 [4] 337 (1964).

The above understood Date and Date

Excess $SrTiO_3$ (Excess 2) (excess 1)

13.531 TiO_2 25.25 $SrCO_3$ 13.6 TiO_2 25.00 Sr
13.53 25.28 13.61 25.02
.03 tloss

zero off after addition probably static

In drying oven @ ~100C under house Vacuum @ 1:30 p.m. 4/6/88

38.72 after drying
87.87 crucible
126.59 w/ addition
118.85
7.74

38.43 after drying
97.07
125.50
117.79
7.71

$25g \times \frac{101.96}{375.14} \times \frac{103}{147} = 17.5$ ~~6.795~~

~~6.795/7.7 = 88.25%~~ 12% excess loss
7.5/7.7 ≃ 97.5% +

#1 ← TiO_2 #2 ← $SrCO_3$

NO GREEN DATA TAKEN → No GREEN DATA

2.75	0.529	0.159		4.96	1.03	3.01	0.517	0.175		5.0	1.04
	1.321	0.404	0.554				1.313	0.444(5)	0.60		
N_2						EXCHANGE					
2.71	0.529	0.160		4.71	97.9	3.00	0.520	0.176		4.89	1.02
	1.344	0.406	0.576				1.321	0.447	0.613		
SiB2						SiB1					
~~2.38~~	0.540	0.135		4.69	97.5	2.56	0.529	0.150		4.74	98.5
	1.372	0.343	0.507				1.314	0.381	0.540		
N_2											
2.41	0.554	0.141		4.31	89.6	2.60	0.526	0.151		4.83	1.00
	1.410	0.358	0.559				1.336	0.384	0.538		

SUBSTITUTION 1 : Al_2O_3 1 mol % 16 HRS RXN.
" 2 : V_2O_5 ↓

	N_2	O_2
1 :	100.4	98.5
2 :	89.6	97.5

EXCESS DOPING 1 : ½ mol % TiO_2
" " 2 : ↓ $SrCO_3$

	N_2	O_2
1 :	97.4	103
2 :	102	104

"MECHANICAL MANIPULATION"

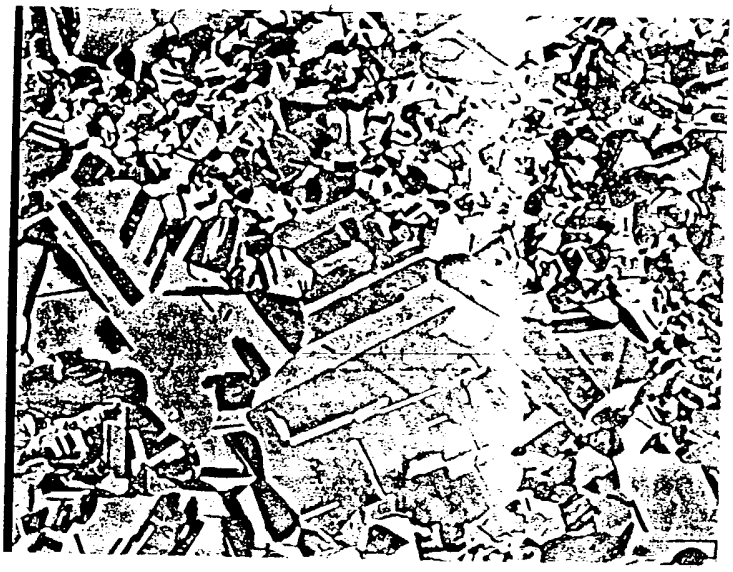
fines : 103.4

fines ⅓, med ⅔ mix : 102.3

MED : 100.4

4/11 Pharr Co/Bi studies

600C overnite N_2 treatment on as rcvd material (Cu)



100 μm

INTERIOR INHOMOGENOUS (ABNORMALOUS) GRAIN GROWTH

100X

100 μm

EXTERIOR ~HOMOGENEOUS

The above understood and witnessed by _____ Date _____ and by _____ Date

1, 3, 10% Bi in copper.
5 g total per batch

4.95 post 750C

gBi : 5(0.01) = 0.05 5(0.03) = 0.15 ~~5~~(0.10) = 0.5 g

	Cu		
	4.95	4.85	4.50
actual	0.05-6	0.15-0.16	0.50-1
	4.95-6	5.00	~~4.99~~-5.0
recovery	4.87 added to 5.00	4.99 —	4.95
	~~5.00~~		
	4.98 after 400C overnite (not disturbed)		490 after 400C overnite

Reloaded 1% & will continue w/ 3%. Will make a new 10% and a 50% and fire @ 750C. Crucible shortage ⇒ will likely modify above.

50/50 2.5g Bi, 2.5g Cu

gBi: 2.51 actual

post Cu 5.01

Cu 2.~~40~~

crucible total 1.34, ~~6.34~~ 5.00 ~~5.00~~

Post 750C 6.3 (4.91)

10% Bi

0.51
5.02(3) total
4.51(2)

1.32(3)
~~6.24~~ ~~5.07~~ ? 4.92

Post 750C 6.21 (4.89) 400C RESULT

3% as above

NOTE: INITIAL 400C (420C) follow (1-10%) percentages of Bi did not produce expected densification/solidification of pucks.

The above understood ... Date ... and ... Date

25 & 35% Bi/Cu melts & 1 Bi crucible filling

25%

0.25(5) = 1.25 g Bi, 3.75 Cu

6.33 pre
1.33
5.00

6.30 post Δ = 0.03

Possible post density: 0.315, 0.385
4.97 0.800, 0.978 0.6

35

1.75 Bi ; 3.25 Cu

6.25(4) loaded
1.30(1)
4.95 starting total

6.23 Δ = -0.02

8.25 90.

definitely smaller volume, higher density

Argon/H2 Bi filling

13.32 post
PRE 25%
1.26 Bi
5.03 w/Cu

filled 12.82
crucible: 11.39(46)
1.43

CRUX => 11.86
0.47

13.30 after slag removal

post 12.81

Argon/H2 25% Bi Run ← Pellet Run →

pellet: 4.87 0.485 0.222 7.25 79%
1.232 0.564 0.672

8.96 × 0.75 + 0.25(9.8) =
6.72 + 2.45 = 9.17

Furnace started 2HR purge 3:35
4.84 0.483 0.234 pellet bloating d/t Bi vaporization?

Bi_2O_3: sp. g. 8.8 m.p. 820°C

The above understood and witnessed by _____ Date _____ and by _____ Date _____

$SrTiO_3$ Grain Growth Experiment - Mechanical Measures

1) PSD weighting
2) fines full density & free sintering of polished surface
3) reacted to constant weight (last batch) sintering as in #2

Fines = F

2.04	0.570	0.155		3.14	65.3
	1.498	0.394	0.649		
2.01	0.488	0.132		4.975	10[illegible]
	1.239(5)	0.335	0.404		

Fine/medium = FM

2.19	0.577	0.154		3.32	69.0
	1.465	0.391	0.659		
2.17	0.504	0.135		4.92	102.3
	1.28	0.343	0.441		

M (chipped)

2.46(7)	0.582	0.165		3.43(5)	71.4
	1.478	0.419	0.719		
2.46*	0.519	0.147		4.85	100.9
	1.318	0.373	0.509		

REMARKS >> * some powder adhered

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Sintering Regime

Rapid Temp w/ 10 cc/min O_2 4:25 p.m.

1550C initial set, after reaching temp for 1 HR, 1640C overnite

5:20 p.m. ~ 1100C, $T_{CONTROL}$ blown off ~ 30-45 minutes @ 1540.

Restarted @ ~ 5:55 & brought directly to 1640C.

6/4-26-88

LEAK TESTING - SEE FORR HIGH VAC

25 millitor after continued pumping through system STARTING WITH Roughing Valve

Will check pumpdown through HVAC valve alone tomorrow.

4/27 → Vacation

4/28 Pump down through high vac initially unsuccessful, must have been stuck valve, but after freeing can get down to ~50 millitor in 15 minutes. Will continue pumping.

1/2 40

Down to 10 thru rough, 30 w/ HVAC only,

Rather quick leak-back when both closed off indicating leaks in system:

1) FURNACE
2) Elbow connection
3) Pump

See page 78

5/13 Promised CSS test Monday (Check!)

5/13

First Milling - Autoseed - Teflon liner C3.

48.17 g known, but "few" added before total processed wght. checked

49.69 g yield

PASS II 5/16

47.6 g yield: much fluffier, looks ~like 5 μm pwdr.
IMMEDIATELY RECYCLING
pg. 66 for PSD sheets ~2g loss!

PASS III 45.8
45.8

PASS IV 44.5
~~18.0~~ 5 pellets
26.5

KORIBA CAPA-5EE
PARTICLE ANALYZER

DATE 5/13
Autoseed SAMPLE C3 - Pass I
SOLVENT ISO
Teflon liner

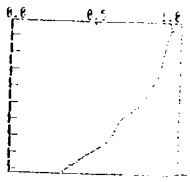
* CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

* TIME 0 H 4 MIN 20 SEC

* DATE

TIME ABSORBANCE



* DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	49.7	49.7
10.0- 9.0	1.8	51.6
9.00-8.00	4.5	56.1
8.00-7.00	2.7	58.8
7.00-6.00	6.1	64.9
6.00-5.00	6.9	71.7
5.00-4.00	10.2	81.9
4.00-3.00	4.0	85.9
3.00-2.00	6.0	91.9
2.00-1.00	5.0	96.8
1.00-0.00	3.2	100.0

D(AVE) 5.65 (PM)

* DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

← High large particle % 47.7 vs 18 for C2

← overall ave. ~10 vs 5-6 for C2
however distribution seems similar { some bypassing must have occured.
Will work on Tuesday.

The above is and witnessed by _____ Date

and by _____ Date

Date and Date

Bi_2/Cu 25/75 crucible packed, sinter overnite in Ar/H_2(5) @ 750C.

#1 ⇒ < ~~100~~ 300 mesh Cu, spherical ("new")

#2 ⇒ ~~100 mesh spherical Cu~~ 10μm (old)

#3 ⇒ penetration 10μm

	#2: 10μm		#1. spherical	
	1.25	5.01	1.25	5.00
	6.24		6.35	
ceux	1.34		1.35	
	5.00		5.00	
POST	6.31		6.32	
	8.0-8.5 mm l		8.37	
	9.65-9.75 mm dia		9.65-10.0	

After interruptions: 5/24 start cut & polishing

6/8/88 finish: 6/8/88 after

{Porosity is reduced, and 3rd ϕ 'oxide' has been eliminated (grey) in forming gas.}

The above understood and witnessed by _____ Date _____ and by _____ Date _____

C3- P1-5 Green Data & Sinter → Picked up mill teflon material ruined

	ISO 275 G	mm	mm			
C3P1	3.59	1.470	~~0.479~~			70.5
	3.33			0.80		
C3P2	3.33	1.477 cm	0.492 cm		4.90	69%
	2.93			0.757		
C3P3	3.58	1.472	0.478	~~E~~ 0.813	4.40	69%
C3P4	3.37	1.476	0.490			
C3P5	3.53	1.478	0.471		5.52	87 even w/ cracking puck OK. I think Mill no good.
	3.19!	1.331	0.415	0.577		

NOTES: PELLETS SEEM TO HAVE SHED ORGANIC/GAS?! Looks like the pellet melted in plastic container.

ISO 26 UNI 6700 20°/→550, 10°/→975, 2HRS;

C3P6	2.52	1.50	0.32	0.565	4.46	70.1%
Coarse	2.50	1.482	0.316	0.55	4.55	71%

The above understood and witnessed by _____ Date _____ and by _____ Date _____

Calcinations - 750C IN @ 8.88 P.M. 5/17/88 out 9:00 AM 5/18 69

TiO_2 - Cmer 39%

16.3620
.8610
15.501 g TiO_2 weighed

89.4610 crux
105.0174 crux + TiO_2
15.5564 g TiO_2 by difference ~~99.64%~~ → 0.3% Δ + 0.0554

5/18 POST
105.0174
104.9395 gaining ΔT > ΔT_{CO_3}
0.0779 / 15.501 = 0.5% +

EQ → 105.0084
105.0174
0.0090 / 0.0779 = 88.5% back

$SrCO_3$

18.4193
0.8752 → 0.8752
17.5473 17.5441 Δ 0.0032 ≈ ΔΔ Dif !!

109.9615
92.3660
17.5955 Δ + 0.0514

ΔΔ Dif ⇒ 0.004g ~ 4mg calibration

POST 109.9615
109.8870
0.0745 / 18.5441 = 0.4% gaining

EQ 109.9510
109.9615
0.0105 85.6% back

5/19 TiO_2 { ~~$SrCO_3$~~ 2nd Cal POST $SrCO_3$

105.0174
104.8670 (-0.0003 cal)
.15 ~1.%

109.9615
109.8580 (-0.0003 cal)
0.1035 ~ 0.6%

The above understood and witnessed by _____ Date _____ and by _____ Date _____

The above understood and witnessed by _____ Date _____

and by _____ Date _____

HF Silicon Etch/Wash/Buffer Solns.

80g NH_4F in 120g H_2O (Distilled) ⇒ ~120cc 40 wt. % NH_4F REAGENT

actual soln is 40:1 ~170cc

BHF ⇒ 40 parts NH_4F reagent : 1 part HF (49 wt%) solv.

QUENCH ⇒ 10:1 DI : NH_4OH reagent 50ml : 500ml

BHF clean ⇒ 10:1:2.2 (NH_4F : HF : Glycerin)
reagent 49

16(10) = 160 16(1) = 16 16(2.2) = 35.2 = 211 ml
320 (55)
350
(190) (77)

MSG:FROM: SARDESAI--FSHVMCC TO: MDT --YKTVMT 05/18/88 12:39:40
To: MDT --YKTVMT

From: Viraj Sardesai
8-533-8545, SCL Pers Metals,GTD E.Fishkill
IBM INTERNAL USE ONLY (Unless otherwise specified)
SUBJECT: BHF concentrations used in SCL

Michael,

We use 40:1 BHF for pre platinum, emitter screen ox removal and for s metal preclean.

The chemical is commercially available premixed solution and has 40 parts (by volume) of 40 wt pct NH4F solution mixed with 1 part of 49 wt pct HF solution. Both NH4F and HF are in aqueous solutions. Manufacturer specs the HF concentration to 0.61 to 0.77 moles per liter and specific gravity of 1.106 .

For S postL/O BHF clean 10:1:2.2 (NH4F:HF:Glycerin) is used prepeared similarly and quenched in 10:1 NH4OH solution (28 Wt pct NH4OH solution diluted to 10 times its volume in DI water).

cc: SZECSY --FSHVMCC
HOUGHTON--FSHVMCC

Regards,
VIRAJ
FSHVMCC(SARDESAI),D/11G B/322 Z/5T1
********** OUR TEAMWORK MAKES THE DIFFERENCE ! **********
.BHF concentrations used in SCL

HF

BHF CLEAN

QUENCH

The above understood and witnessed by _____ Date _____ and by _____ Date _____

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...ered IBM Confidential"
...egister with local Recorder

Date and si... ...ry entry. Have every possibly important entry w... ...Submit an Invention Disclosure of anythin... ...sibly new and inventive.

The above understood and witnessed by _____ Date _____

and by _____ Date _____

5/18 Polymilling

Pass	40g batch	YIELD	PSD 1	2	Δ
I		36.8	6.94	4.84	3
II		33.4	3.65	3.21	.3
III		31.3	3.53		(2) bag changes required
IV		28.4	2.98 !		3 actually higher erroneous 7% @ 410
V		25.75	3.1 !		2.5 New bag

NO

The above understood and witnessed by _____ Date _____ and by _____ Date _____

ISO 28,

MARGINAL GREEN DENSITIES

C3P6	3.29	1.474	0.471		4.11	64.6
	3.23	1.343	0.416	0.80 0.59	5.48	86.2 !
C3P7	3.19	1.476	0.455		4.00	64.5
	3.13	1.343	0.404	0.779 0.57	5.49	86.3
C3P8	3.21	1.473	0.467	0.787		64.1 } TO KING-TU
	3.16	1.341	0.409	0.577	5.48	86.2 }
C3P9	3.08	1.474	0.441		4.09	64.3
	3.02	1.336	0.391	0.7525 0.548	5.51	86.6

NOTES: Pellets in @ 4:55 with flowing O_2 (bottled, dessicated)
Heating started @ 5:15 @ 20°/min (97C @ start)
@ 265C cut back to 10°/min; to reach sinter T° @ 6:35 p.m.
Sintering @ 975C for 2 HRS. till 8:35 p.m.
Quench & remove.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

995C sinter

CSP10	3.18	1.474	0.453	0.773cc	4.114	64.7
	3.11	1.338	0.399	0.561	5.54	87.2

no (appreciable) lia. ∅ !

T3 "try"

yield	PASS	Δ		Ave. part dia, <100 mesh
10.35	0			
29.~	I	TO !	1-2g leaks	3.29
21.5	II	8 !	~1g leak	3.11
18.5	III	3 !	~~4.0~~ g of ~2.0μm pwde in mill neck	

22.4 total mix yield

The above understood and witnessed by _____ Date _____ and by _____ Date _____

The above understood and witnessed by _____ Date _____

and by _____ Date _____

C3P10-18							
975C → C3P10	①	3.14	1.461	0.473		3.975	62.50
		✓3.08	1.308	0.400	0.79	5.75	90.4
					0.536		
C3P12 C	②	3.28	1.462	0.495			
		✓3.21	~~1.308~~	0.417	0.56	5.73	90.0
C3P13	③	3.04	1.463	0.450			~~89.31~~ ← RECHECK TOMORROW
		2.97	~~1.307~~ 1.310 1.306	0.388 0.385	~~0.54~~ 0.523 0.516	5.68 5.756	90.5
C3P14		3.21	1.464	0.473			
		✓3.14	1.308 1.306	0.405 ✓	0.548	5.77-8	90.7-9
1000C → C3P15		3.04	1.461	0.458			
		2.97	1.298	0.386	0.511	5.81	91.35
C3P16 (polished)		3.08	1.466	0.457			
		3.02	1.301	0.390	~~0.58~~ 0.519	5.82	91.5
C3P17		3.28	1.464	0.492		~~5.75~~ 5.88	90.4 → ~~91.5~~ chip off ~~edge~~
		3.22 (3.28)	1.298	0.422	0.558		

The above understood and witnessed by _____ Date _____ and by _____ Date _____

IBM Technical Notebook

6/8/88

Centorr HVAC system close to finished with leak testing, preliminary operation checks.

System mechanical pump down < 20-30 minutes

Turbomolecular ↓ down → to 5×10^{-5} within 1 hr with attainment at mid 6's by 1:00 p.m.
Start was 9:15 originally.

Block-off flanges, new Centorr purge & plug fittings still needed. Follow leak test reqs.

Bi/Cu Free-Crucible Sinter Vacuum Run

To glass shop 6/7/88. Batch size to be 4.0 g to allow for ease of manipulation during sealing of quartz tube.

4.0 (0.25) = 1.00 g Bi

↳ (0.75) = 3.00 g Cu (will use 10 μm Cu powder)

Bi → 1.00 (0.99)
Cu to 4.03
Cu 3.03

from JAR 4.02 (1)

cruc 1.33
w/ mix 5.33 (4)
4.00 (4)
↳ 4.01

~ 24.8 %
75.2 %

~ 25
75

Redo - spilled in glass shop

6/9 New crucible shape/size for stability

Lets take 5.0 g batch

1.25/5.0 (±0.01)

crucible 3.35
w/ mix 8.35
5.00

6/14 After overnite sinter & removal from quartz tube

cruc & sinter 8.30 g (some spillage in tube before heat treatment)

no appreciable vapor product stream

Conclusions: Vacuum doesn't appear to work as well as Ar/H2. Sample full of holes, but no evidence of ablation, so holes are real. Again, no evidence of vapor phase deposition in tube.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

IBM Technical Notebook

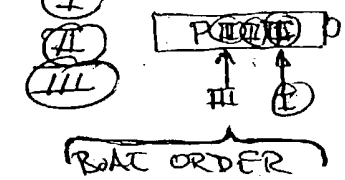
6/14/88

To do: 10, 20, 25 % in Ar/H_2 (5) ← MONDAY (VACATION)

RUN !

WEDS 6/22/88

5 gram batches: 0.25 (5) = 1.25 Bi / 3.75 Cu
0.20 (5) = 1.00 Bi / 4.00 Cu
0.10 (5) ~ .5 Bi / 4.5 Cu



(I) 5 gram w/ new conical crucible
mix 9.03
tare 4.03
5.00
↑
PRE

voids, but some areas seem OK.
less small voids, some good regions versus (II),
however large voids & inhomog. Need repeating, maybe longer times.

(II)
9.00
tare 4.00
5.00

seems very good, no large voids, mix exam next, microscope exam should show many small "pockets" or voids. Usually circular.

(III) "old"
6.31
tare 1.31
5.00 'nominal loss possible'

did not densify fully

Administrative Note

$SrTiO_3$ Milling Results

HORIBA CAPA-500
PARTICLE ANALYZER

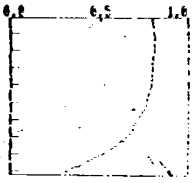
DATE 3/24/88
SAMPLE $SrTiO_3$-3
SOLVENT 150
MI-MED

* CONDITIONS SLIGHT IMPROVEMENT

SOLV.VISC	2.16(CP)
SOLV.DENS	6.79(G/CC)
SAMP.DENS	4.81(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

* TIME 0 H 6 MIN 0 SEC
* DATE D= 0.8

TIME ABSORBANCE

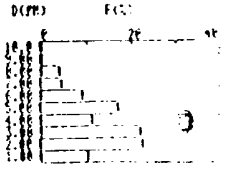


* DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)	
16.0 <	0.0	0.0	✓
16.0- 9.0	0.0	0.0	✓
9.00-8.00	0.0	0.0	✓
8.00-7.00	3.9	3.9	
7.00-6.00	4.4	8.4	
6.00-5.00	8.9	17.2	
5.00-4.00	17.1	34.3	
4.00-3.00	11.1	45.4	
3.00-2.00	22.0	67.4	
2.00-1.00	22.5	89.8	
1.00-0.00	10.2	100.0	

D(AVE) 2.79 (PM)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

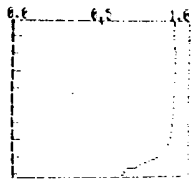
DATE 3/21/88
SAMPLE $SrTiO_3$-DRC2
SOLVENT 150
MIII-5

* CONDITIONS

SOLV.VISC	2.16(CP)
SOLV.DENS	6.79(G/CC)
SAMP.DENS	4.81(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

* TIME 0 H 6 MIN 0 SEC
* DATE

TIME ABSORBANCE

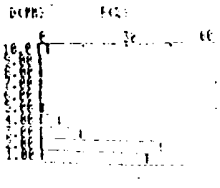


* DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
16.0 <	0.0	0.0
16.0- 9.0	2.9	2.9
9.00-8.00	0.0	2.9
8.00-7.00	0.0	2.9
7.00-6.00	0.0	2.9
6.00-5.00	0.0	2.9
5.00-4.00	0.0	2.9
4.00-3.00	5.9	8.8
3.00-2.00	13.4	22.0
2.00-1.00	41.0	64.0
1.00-0.00	35.9	100.0

D(AVE) 1.34 (PM)

* DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

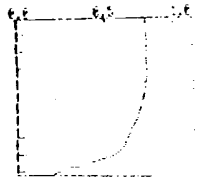
DATE 3/24/88
SAMPLE $SrTiO_3$-DRC2
SOLVENT 150
MIII

* CONDITIONS

SOLV.VISC	2.16(CP)
SOLV.DENS	6.79(G/CC)
SAMP.DENS	4.81(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

* TIME 0 H 6 MIN 0 SEC
* DATE

TIME ABSORBANCE

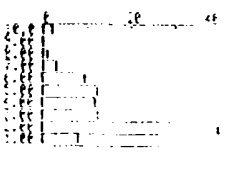


* DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
16.0 <	0.0	0.0
16.0- 9.0	1.0	1.0
9.00-8.00	0.0	1.0
8.00-7.00	1.0	2.0
7.00-6.00	2.9	5.0
6.00-5.00	5.0	10.0
5.00-4.00	12.5	22.5
4.00-3.00	12.1	35.0
3.00-2.00	12.0	50.0
2.00-1.00	39.0	89.0
1.00-0.00	7.9	100.0

D(AVE) 2.20 (PM)

* DISTRIBUTION GRAPH (BY VOL.)



Administrative Notes

C3 Milling RESULTS – T1 = Teflon T2 – Poly W T3 – Poly W

T1

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PIV
SOLVENT 150

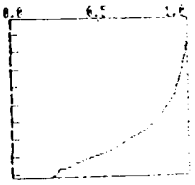
• CONDITIONS T1

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

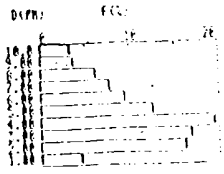


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	3.3	3.3
9.00-8.00	3.6	6.9
8.00-7.00	6.1	13.0
7.00-6.00	7.7	20.7
6.00-5.00	9.5	30.2
5.00-4.00	12.6	42.8
4.00-3.00	19.6	62.4
3.00-2.00	16.8	79.2
2.00-1.00	16.2	95.4
1.00-0.00	4.6	100.0

D(AVE) 3.63 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PIII
SOLVENT 12

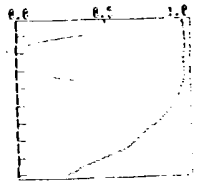
• CONDITIONS D≈0.97

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

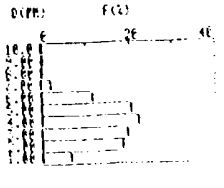


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	2.1	2.1
6.00-5.00	11.5	13.6
5.00-4.00	26.2	33.8
4.00-3.00	21.9	55.7
3.00-2.00	19.4	75.1
2.00-1.00	18.3	93.4
1.00-0.00	6.6	100.0

D(AVE) 3.26 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-PII
SOLVENT 150

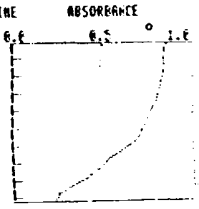
• CONDITIONS D~0.9

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

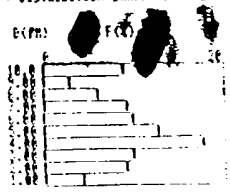


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	9.9	9.9
10.0- 9.0	8.9	18.8
9.00-8.00	2.8	21.6
8.00-7.00	6.0	27.7
7.00-6.00	9.2	36.8
6.00-5.00	9.8	46.6
5.00-4.00	12.6	59.2
4.00-3.00	17.6	76.9
3.00-2.00	9.8	86.7
2.00-1.00	9.0	95.8
1.00-0.00	4.2	100.0

D(AVE) 4.73 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



Administrative Notes

T2 Part I

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

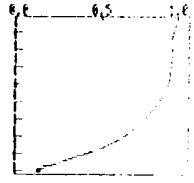
• CONDITIONS

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.9

TIME ABSORBANCE



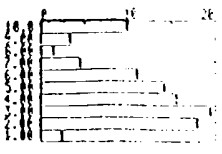
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	5.8	5.8
9.00-8.00	3.4	13.3
8.00-7.00	1.5	14.8
7.00-6.00	4.5	19.2
6.00-5.00	11.2	30.4
5.00-4.00	14.1	44.5
4.00-3.00	15.6	60.1
3.00-2.00	19.4	79.5
2.00-1.00	18.0	97.5
1.00-0.00	2.5	100.0

D(AVE) 3.65 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

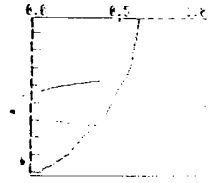
• CONDITIONS

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.57

TIME ABSORBANCE



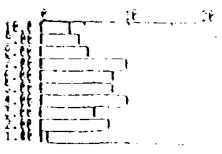
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	30.4	30.4
10.0- 9.0	3.2	33.6
9.00-8.00	4.3	37.9
8.00-7.00	5.5	43.4
7.00-6.00	9.8	53.2
6.00-5.00	6.2	59.4
5.00-4.00	8.1	67.5
4.00-3.00	9.5	77.0
3.00-2.00	6.2	83.2
2.00-1.00	7.8	91.0
1.00-0.00	8.7	99.7

D(AVE) 6.94 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-PI-T2
SOLVENT 150

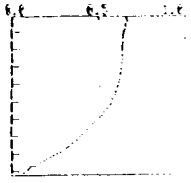
• CONDITIONS

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~.67

TIME ABSORBANCE



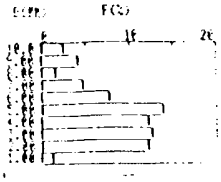
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	27.1	27.1
10.0- 9.0	2.5	29.6
9.00-8.00	4.1	33.7
8.00-7.00	1.5	35.2
7.00-6.00	4.6	39.9
6.00-5.00	7.9	47.8
5.00-4.00	13.8	61.6
4.00-3.00	12.3	73.9
3.00-2.00	12.7	86.6
2.00-1.00	12.2	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



Administrative Notes

T2 PART 2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-P4-T2
SOLVENT 150

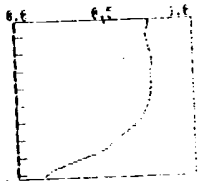
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.7

TIME ABSORBANCE

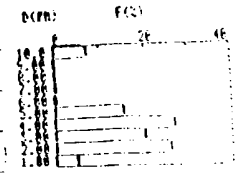


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	7.2	7.2
9.00-8.00	0.0	7.2
8.00-7.00	0.0	7.2
7.00-6.00	0.0	7.2
6.00-5.00	0.3	7.6
5.00-4.00	15.1	22.7
4.00-3.00	27.0	49.7
3.00-2.00	20.0	69.7
2.00-1.00	25.6	95.3
1.00-0.00	4.7	100.0

D(AVE) 2.56 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19
SAMPLE C3-P3-T2
SOLVENT 150

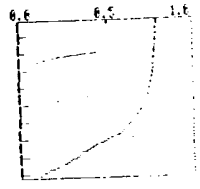
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.7

TIME ABSORBANCE



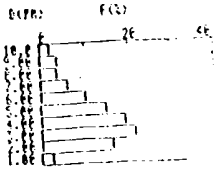
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	2.1	2.1
9.00-8.00	3.9	6.0
8.00-7.00	3.7	9.8
7.00-6.00	6.2	16.0
6.00-5.00	10.1	26.0
5.00-4.00	15.0	41.0
4.00-3.00	19.6	60.7
3.00-2.00	21.4	82.1
2.00-1.00	16.0	97.9
1.00-0.00	2.5	100.0

D(AVE) 3.53 (PM)

NO MILLING / BAG CHANGE

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/19/88
SAMPLE C3-P2-T2
SOLVENT 150

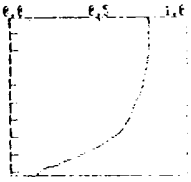
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

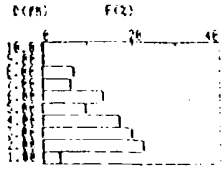


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	7.6	7.6
7.00-6.00	6.4	13.4
6.00-5.00	13.4	26.8
5.00-4.00	9.8	36.6
4.00-3.00	17.1	53.6
3.00-2.00	20.0	73.6
2.00-1.00	22.4	96.0
1.00-0.00	4.0	100.0

D(AVE) 3.21 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



Administrative Notes

T2 (cont.)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE _____
SAMPLE S3-P5-T2
SOLVENT _____

• CONDITIONS

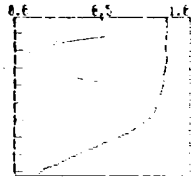
SOLV.VISC 2.18(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.87

TIME ABSORBANCE



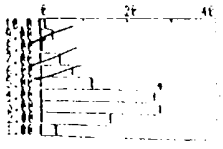
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.0	1.0
9.00-8.00	2.0	3.0
8.00-7.00	8.1	4.0
7.00-6.00	4.4	8.4
6.00-5.00	7.3	15.8
5.00-4.00	11.8	27.5
4.00-3.00	26.1	53.7
3.00-2.00	27.3	81.0
2.00-1.00	15.0	96.0
1.00-0.00	3.4	100.0

D(AVE) 3.14 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 9/19
SAMPLE S3-P5-T2
SOLVENT 150

• CONDITIONS

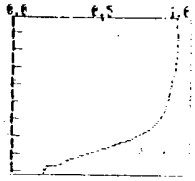
SOLV.VISC 2.18(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 10.0 (PM)
D(MIN) 1.00(PM)
D(DIV) 1.00(PM)

SPEED 500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~0.9

TIME ABSORBANCE



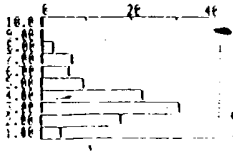
• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	2.3	2.3
7.00-6.00	6.8	9.1
6.00-5.00	6.6	15.6
5.00-4.00	9.6	24.6
4.00-3.00	22.5	47.1
3.00-2.00	30.6	77.8
2.00-1.00	18.0	95.7
1.00-0.00	4.3	100.0

D(AVE) 2.91 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)



Administrative Notes

T1 versus T2 versus T3 for VARIOUS PASSES

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/23
SAMPLE C3-P5-T3
SOLVENT 150

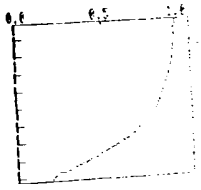
• CONDITION T3

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA ~ 0.9

TIME ABSORBANCE

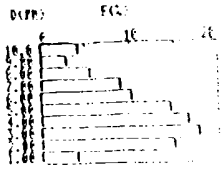


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	4.3	4.3
9.00-8.00	2.9	7.2
8.00-7.00	5.5	12.7
7.00-6.00	9.0	21.7
6.00-5.00	10.2	31.9
5.00-4.00	14.6	46.5
4.00-3.00	16.6	63.1
3.00-2.00	17.9	81.0
2.00-1.00	15.0	96.0
1.00-0.00	4.0	100.0

D(AVE) 3.79 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 5/17
SAMPLE C3-P1-T1
SOLVENT

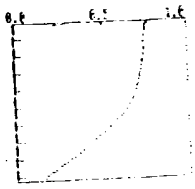
• CONDITIONS 0.75

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA T1

TIME ABSORBANCE

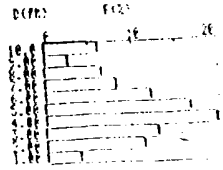


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	6.2	6.2
9.00-8.00	2.7	8.8
8.00-7.00	6.6	15.4
7.00-6.00	8.2	23.6
6.00-5.00	12.0	35.6
5.00-4.00	16.8	52.4
4.00-3.00	19.5	72.0
3.00-2.00	12.8	85.0
2.00-1.00	11.2	96.3
1.00-0.00	3.7	100.0

D(AVE) 4.14 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE
SAMPLE C3-P1-T2
SOLVENT

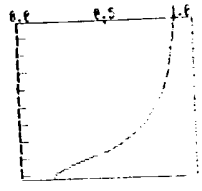
• CONDITIONS NO CHANGE
MILL CONFIG REQUIRED

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	16.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA T2

TIME ABSORBANCE

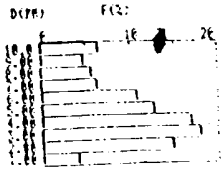


• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	6.5	6.5
9.00-8.00	4.7	11.2
8.00-7.00	5.6	16.8
7.00-6.00	6.3	23.1
6.00-5.00	10.7	33.7
5.00-4.00	12.7	46.4
4.00-3.00	16.9	63.4
3.00-2.00	18.0	81.4
2.00-1.00	14.7	96.1
1.00-0.00	3.9	100.0

D(AVE) 3.79 (PM)

• DISTRIBUTION GRAPH (BY VOL.)



Administrative Notes

FINAL C3-T3- RESULTS : NECK, MIX & premix medium

HORIBA CAPA-5EE
PARTICLE ANALYZER

SAMPLE
SOLVENT

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

NECK

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <		
10.0- 9.0		
9.00-8.00	0.0	0.0
8.00-7.00	2.4	2.4
7.00-6.00		4.4
6.00-5.00	10.5	14.9
5.00-4.00	13.1	28.0
4.00-3.00	6.5	34.5
3.00-2.00	17.0	51.5
2.00-1.00		87.4
1.00-0.00		100.0

D(AVE) 2.89 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40

10.00
9.00
8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00

HORIBA CAPA-5EE
PARTICLE ANALYZER

DATE 5/23
SAMPLE C3-T3 MIX
SOLVENT T32

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA D~0.9

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <		0.0
10.0-		1.2
9.00-8.00	1.2	1.2
8.00-7.00	0.0	1.2
7.00-6.00	0.0	1.2
6.00-5.00	0.0	1.2
5.00-4.00	9.7	10.9
4.00-3.00	27.7	38.6
3.00-2.00		65.4
2.00-1.00		52.7
1.00-0.00		100.0

D(AVE) 2.63 (PM)

O.K.

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40

10.00
9.00
8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00

HORIBA CAPA-5EE
PARTICLE ANALYZER

DATE 5/25/88
SAMPLE C3-T2-T3
SOLVENT T50

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	1.00(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 26 SEC

• DATA

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	1.8	1.8
9.00-8.00	0.0	1.8
8.00-7.00	3.1	4.9
7.00-6.00	4.3	9.2
6.00-5.00	9.4	18.6
5.00-4.00	12.8	31.5
4.00-3.00	20.7	52.2
3.00-2.00	30.0	82.2
2.00-1.00	14.3	96.5
1.00-0.00	3.5	100.0

D(AVE) 3.11 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

0 20 40

10.00
9.00
8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00

ATTACHMENT C

90 10179

IBM

Technical Notebook

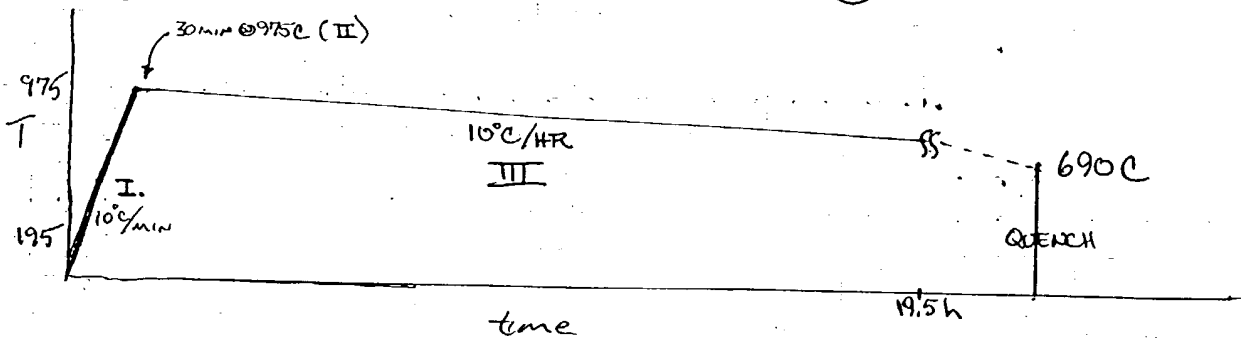
Book V

User's Initials and Last Name: DUNCOMBE, P.		
Employee Serial: 155139	Date of First Entry: 6/7/88	Date of Last Entry: 5/89

Security Classification:

6/9/88

Next "transition" Heat Treatment (B)



I. 25C-975 → Δ950C/10C/min = 1.583 h (1h 35min)

II. 975C for 30 min = 0.5 h

III. 975C - 690C = Δ285C/10°C/h = 28.5 h

total: 28.5 + 0.5 + 1.58 = 30.58 h

Proposed: purge till 10 A.M. Monday
10 - 11:35 AM. → heat up (RAMP 1)
11:35 - 12:05 pm → dwell
12:05 pm - 4:35 p.m. Tuesday → RAMP 2 (cool)
4:35 p.m. Tuesday quench

(A) As B above w/ 650C QUENCH w/ I: as above
II: as above
III: 32.5 h
} ~34.5 h total

ON @ 11:10 A.M. 6/13
START RAMP down @ 1:45 p.m. (actually 2:05)
Drop to 946 @ 10C/min then slo RAMP to 0.17C/min

Estimated Quench time 25h 24min OR 3:36 p.m. 6/14/88

6/11/88 ⇒ ~~2:45~~ updated QUENCH TIME 3:15

The above understood and witnessed by _____ Date _____ and by _____ Date _____

fidbit: (conversion)

mmHg = 0.001333 bars ∴ 0.001333 bar/torr
↓ = 1 torr

1.333 mbar/mtorr → 0.75 mtorr/mbar

{100, 10°/min, 1550, 61 min, 10°/min, 100C} RUN SPECS 6/29/88

T	PV	PROG 100	MAN%	Amps	Volts	gaseter 6.0	MFC ASP	Comments START
12:20	986	990	10.4	490	2.5	6.92		4×10^{-5} tor
12:42	1196	1200	14.9	600	3.1	7.11		5×10^{-5} torr
12:57	1346	1350	20.1	(700)	4.0	7.27		4.6 X / 12 psi
1:07	1445	1450	25	(725)	4.25	~~7.37~~		3.5
1:17	1550	1547	30.3	700	5.4	7.48	60.1	↓ 12
1:48	↓	1550	29.7	700 700	5.4	7.47	30	1.6 / 12
2:18	1550	1550	29.2	690+	5.25	7.469	0	1.25 / 3
2:32	1415	1407	20.3	625	4.25	7.34	X	8×10^{-6} / 2
3:45	679	678	3.6	300	1.5	6.61	–	$2.8E-6$ / 2
4:20	333	325	0	0	0	6.29	X	2.8 / ✓
4:45			0	0	0			

The above understood and witnessed by Date and by Date

4/7/5/88 STBX-2 **IBM Technical Notebook**

Time	PV	Prog	Mon%	A	V	gauge?	ΔSP	Comments	Vac
10:40	341	341	3.4	350	1.5	7.76		bias changed @ start	
	640	641	5.2	390	1.6	7.98		20 PSIG applied	6×10^{-5}
11:42	962	961	86	460	2.4	8.20		40 ↓ ↓	$1.8E-5$
1:00	1650	1650	35.4	735	6.0	8.88	112.0	✓	$1.9E-5$
2:30	↓	↓	35.3	725	↓	8.86	22.8	∴ 2:52	$2.5E-5$
3:00	1576	1565	28.0	700	5.5	8.78	—	→ 2:53	$1.8E-5$
4:46	532	530	2.5	360	1.7	7.87	— 1.6E-6	load removed (3 PSIG)	$4E-6$
5:07	~300	~300	0.8	<300	<1	7.70	→	increase flow to 8.5 sccm from 7	
5:10								STOP STP	1.6E-6
5:15	200	228	off	—	—	7.61		SHUT OFF HZP	
5:20	149	162	—	—	—	7.52		vacuum constant ~12.5 PV/min	
								SHUT OFF STP Power, started backfill (5:22 STP on mech gears, light out), mech shut down, high vac closed.	
5:25	178	134						GAS flow RAISED temp, but Ar flow GPM up to 18.	
5:30	190	100	Prog off						

Notes: 300C @ 10:36 ∴ 2h 15m to S.P. ⇒ 12:45 (est) + 2h ⇒ 2:45 RAMP down, ~3.5 hrs for cooling, <100C opening @ 6:15 approx (due to thermal mass lag)

Unsuccessful. Some sticking to bottom foil. Cracked with diffusion zone and multiple phase boundary.

6/28, Bi/Cu

Ar/H_2 Bi 20 { Bi 25 pieces remaining after slicing isostatically pressed to 28,000 PSI OK

Bi 25 → some obvious large void improvement on at least the 1/2

Bi 20 → possible visible evidence of compression, need to section. (especially large 1/2)

Objectives:

1) isopress as above

2) slice

3) anneal remaining sections

4) study wetting/densification vs temperature relationship

6/7/7 STBX-3

Time	TV	Prog	M%	A	V	quota	ΔSP
~11:00	462	462	35	350	1.5	7.69	
11:58	1064	1066	9.2	490	2.5	8.23	
11:08	1576	1575	29.5	675	5.5	8.82	22 / 16.3
1:25	1575	1575	29.3	↓	↓	8.81	91.6
2:45	1575	1575	29.2	675	5.5	8.80	15
4:45	599	599	0.0!				

Comments
est. S.P. start: 1:17, 5E-6
1.8E-5
↓ est. ~ 3:00 start sampling
1.0E-5 ⇔ 9.6E-6
7.5E-6

different than last run, even on 7 gpm!

7

7/11 2050 Bake out Pre Al_2O_3 : I88

Time	PV	Prog	M%	Amps	V	gauge	ΔSP MIN	Vac	Comments
12:30	1660	1687	40.1	760	6.4	—	—	5E-5	RAMP 20°/MIN ∴ 1200C/h Some degassing >1650
12:42	1893	1935	66.2	900	8.4	—	—	4E-4 !	7gpm
12:52	2037	2050	88.7	950	9.5	—	10/5	7E-4	definitely baking out top was 2090 [illegible]
12:55	2028	2003	84.4	✓	✓				Hold to continue B-out
1:00			92.0						
1:04	2061	H	96.0					4E-4	Raised manually
1:11	2083	H	98.0						PV steady ↓ ↓ after
1:17	2094	H	↓	1000	10	—	—	3E-4	↓
1:34		same						1.9E-4	Holding
2:05	2092	same						9E-5	Vac increasing as hoped
2:30	2093	switched back to Auto						6E-5	Start PV ramp down
	2093	2000							
2:46	1727	1691	38.9	890	7.5	—	—	2E-6	Good Vac for 1800C RUN
3:20	1004	1000	6.7	475	2.5	—	—	9.8E-7	
3:45	522	520	1.0	200	0.0			8E-7	
4:10	255	off	—	—	—	STP300 off		7.5E-7	up to 11gpm from 7
4:30	138							3.5E-4	STP shut down to melt bearings
4:33									
4:38									

Argon flush after ion shut off

mechanical off

The above understood and witnessed by _____ Date _____ and by _____ Date _____

The above understood and witnessed by

Date

and [illegible]

Date

SET-UP for I88 Al_2O_3 BXSTAL

SEG	T (°C)	time duration	Ramp Rate (°C)	SEG time	Psig
01	100°C	0.1		~~1h 50m~~ 3h	apply initially: 100
02			+600/h	~~2h~~ 5.0m	
03	1800C	5.1	5667	5h	
04			60 - ~~12~~ 80	6.7h	
05	1400			160. ~~24~~ h	remove load @ start ramp
06	100C	0.1	80	16.25	21.25
07	100C				NET ~~25~~ h save 23 total

Time factors: start @ 10:00 A.M., after loading & pumping
Seg 04 begins ~5:00 p.m.
Seg 05 ends ~5:30 p.m. next day O.K.

Pressures from 2/13/87 (Book II pg 49 notes)

$$P_s = \frac{A_{ram(ref)}\,(P_g)}{A_{spec}}$$: ref - ram x section (4.9") P_g - gauge spec - specimen

Psig of 180 for Bxstal Run 87III resulted in yield of moly stage and xstal indentation. So, in lieu of carbon stage switch not possible at moment, will approx. halve pressure to 100 psig and apply load initially, releasing after Ramp down begins. This worked fairly well with Dranes $SrTiO_3$ and similar 87III run yielded only partial cracking of sample.

$$P_{sample} = \frac{(4.9)(100)}{(0.221)} \cong 2,\cancel{440}\ 2,225$$ versus article (ref) max of ~4200 (60%)

The above understood and witnessed by _____ Date _____ and by _____ Date _____

10/7/14 I88 Bxstal Run

Final Prog Seg	T °C	t duration	°C Rate	seg time	Psig	
01	100°C	0.1h		6min	110	
02			60/h	2.8h	110	
03	1800C	4.75h	-	4.75h	110	1.4-4.0E-5 torr
04			80/h	21.25h	Nom.	NOTE: intermed rate 60/h ramp skipped after, however temp fell from ~1790 to 1730 in switchover before 80/h ramp kicked in. 4E-5-8E-7
05	100C					

Run notes: @ ~7:00 pm 7/13 TC leak noticed during inspection. Repaired as possible. Seems to have taken up. See note above.

Results: Severely cracked xstal. Some sticking, but no RXN. with Mo shim on stage, but carbon/Mo RXN from top RAM/strain couple. Pitting/roughing top surface / likely initiating cracks.

WRAP-Up George's Bi/Cu work

Try 10g batch

2.5g Bi 7.5g Cu use

2.48 tare
12.49 after transfer

Also took ½ 20{25 Bi Ar/H₂(5) RUN isostatically pressed forms AND will ANNEAL while SINTERING bar,

To temp @ 3:00 p.m. 7/25/88 18h → 9 A.M. 7/26/88

4:20 p.m. 7/25/88 → new type D {W/25Re/W3Re} thermocouple seems stable AS well as 818 programmer @ 750C

9.99(8) 4.060

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. _____ DATE 7/26/88
SOURCE 25Bc/Cu Bar OPERATOR PRD
TOTAL WEIGHT 13.981 g. OUTGASSING CONDITIONS _____
TARE WEIGHT 4.060 g. _____
SAMPLE WEIGHT 9.921 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_C 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

P_2/P_3 - 3.5415 3.5429 3.5419

DATA

	RUN 1	RUN 2	RUN 3
P_2	19.457	19.656	19.746
P_3	5.494	5.548	5.575
V_P	1.2006 cc	1.219 cc	1.206 cc
DENSITY	8.2634 g/cc	8.138 g/cc	g/cc
	8.26/9.17 = 90%	8.14/9.17 = 89%	8.23/9.17 = 89.75

10A

Average: 89 + 89.75 ≈ 89.5 Between 89-90%

Liq. ϕ Bi/Cu System Development Summary

A. Initial compositions:

% Bi (liq)	% Cu (solid)	Conditions: 400C in N_2 overnite
1	balance	
3		
10		

NOTES: CONDITIONS AND % comp. Bi did not lead to densification (sintering) of plugs. No wgt loss. 5g samples in nitrogen carbon crucibles

B.

% Bi	% Cu	Conditions: 750C in N_2 overnite
50	balance	
10		
(3)?		

NOTES: melting in N_2 does not seem to be total, possibly oxide films influencing behavior. 50/50 mix too much liq. Blue packets apparent. 10% too little liq. No appreciable wgt loss

C.

% Bi	% Cu	Conditions: 750C in N_2 overnite
25	bal	
35		

NOTES: ~density 25 Bi (90) by GA. for calc. (assume 8.9g/cc for 25% fully dense body).
Definitely beneficial liquid phase densification done.
35% Bi density not calculated but plugs obviously smaller ∴ sample density is at least same or higher. Still evidence of incomplete melting. More than 35 Bi evident of possible oxide?

D.

~~Compressed pellet run~~ % Bi	% Cu	Conditions: 750C in Ar/5% H_2 overnite
25	bal	

NOTES: ~calc density 25 Bi pressed (79%) above basis. Evidence of better melting, sintering and densification still prevailed however density by ϕ pellet blasting probably trapped air in plug pores. No significant Bi evaporation evident by wgt loss

E.

% Bi	% Cu	Conditions: 'packed' pyrex crucible "free" sinter @ 750C overnite in Ar/H_2. No compaction.
25	balance	

NOTES: Porosity is reduced, 3rd ϕ (grey) oxide has been eliminated on forming ϕ, wetting seems approx complete, little difference between ~100 mesh spherical Cu and 10μm Cu; though 10μm seems to give better overall results, ~~Centrifuge RUN~~ seems to need more time, but wetting characteristics seem better (will rerun sample in next run (~ 2x overnite))

F. Vacuum 25 Bi Run

conclusions: highly porous. Vacuum doesn't appear to work well ~~in Ar/H2~~. No evidence of oxidation, however, indicating vacuum was O.K. No evidence of vapor phase deposition in glass tube.

G.

% Bi	% Cu	Conditions: 'packed' pyrex crucible sintered @ 750C overnite in Ar/H_2. No compaction.
10	balance	
20		
25		

Results: Good wetting and sintering with 20 & 25% samples, however 20% seems better overall with small uniform pores whereas 25% has some voids as well as pores.

20 & 25% Bi samples pressed to 27,500 psi isostatically. Some collapse of large porosity/voids. Little effect on small porosity.

Annealing effect: 0.02 & 0.09g of Bi-eutl liq. ran out of microstructure upon annealing. Porosity diminished. Very good looking virtually dense 20% sample.

'Base': 25% Bi (10g batch) in pyrex boat first placed in horiz. rocking (classification) then sintered overnite per std. treatment producing very good looking microstructure with little porosity.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

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Date and sign every entry. Have every possibly important entry witnessed. Submit an Invention Disclosure of anything possibly new and inventive.

The above understood and witnessed by

Date

and

Date

8-11-88

GREEN phase - substrate work

ref not found from memory

have one REMAINING substrate, ~80-90% dense, single phase, sinter T 1350C

① pressed 0.2", 0.2g pellet of eutectic

8-18-88 week summary

211 coarse "off comp"

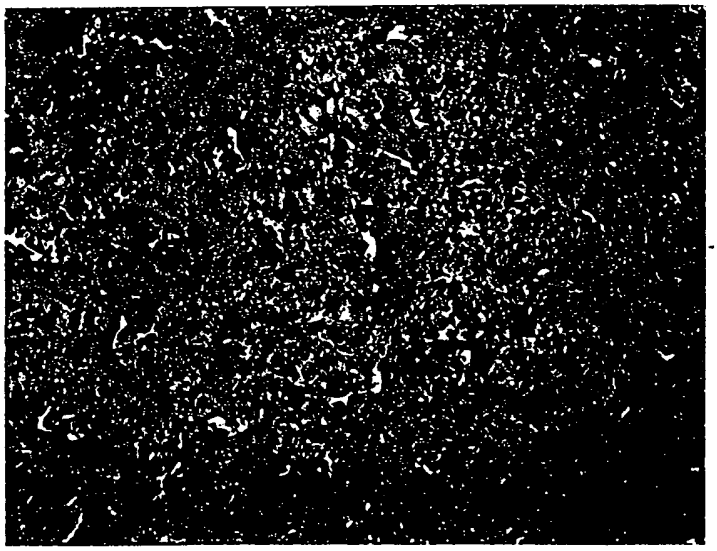
1500C pellet almost totally melts (2ϕ) with interaction between Al_2O_3 and liq. ϕ.

1400C pellet RETAINS ITS INTEGRITY, BUT LARGE AMOUNT of LIQ forms 2ϕ, INTERACTION w/ liq ϕ AND support

1350C liq ϕ still present, though diminished. less interaction for short sinter time

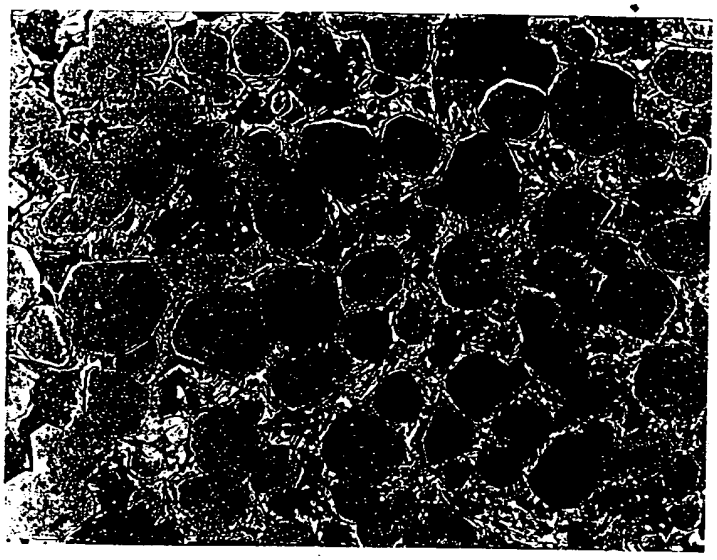
211 milled "on"

1300C 211 1 H 100X milled powder.

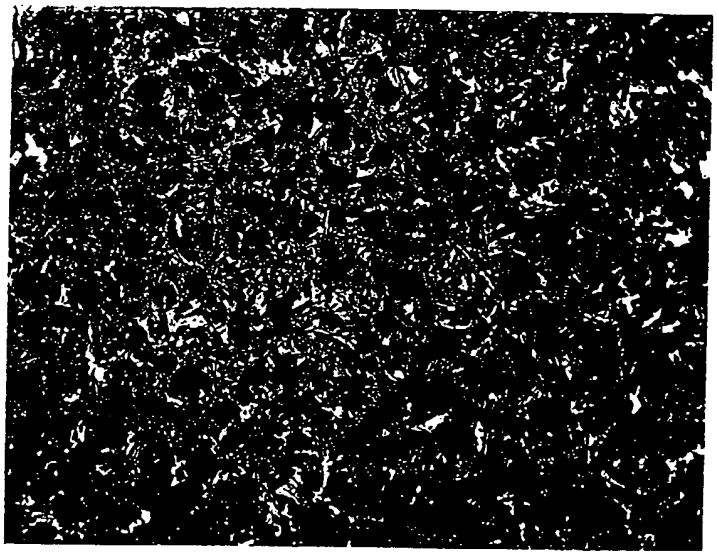


The above understood and witnessed by _____ Date _____ and by _____ Date _____

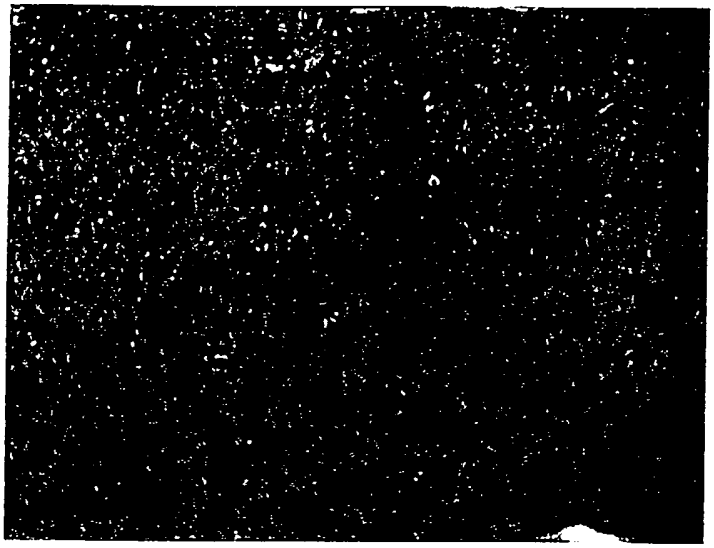
1292C 18HRS 211 milled 100X



1265C 'coarse' 'off comp' OVERNITE 100X



211 milled 1235C 2HRS 1000X



Conclusion: sintering @ 1292C or higher creates 2 Ø material exaggerated 1Ø Ø grain growth after prolonged period

sintering @ 1235C does not induce adequate sintering. pellet REMAINS green as opposed to higher temp, where pellet turns black (presumably thus is not simply surface effect, but has chemical basis)

SINTERING @ 1265C MAY be optimal.

Push definately ooo?

Green density - 211m 0.6 pellet

5500

2.56 1.548 0.399 0.7509 3.41 / (6.36) = ~50%

150

✓ 1.472 0.389 0.662 3.87 / (6.36) ~ 60.9

(6.00) ~64.5

Post ⇒ pellet not good enuf to bother. 2ϕ, stuck, etc. (1292C)
18H

1265C pellet II (ISO 29)

2.56 1.457 0.398 0.66 3.88 / (6.00) ~ 64.5

pellet cracked on checking must REDO, Temp O.K. though (Grain 5C)

pellet III in furnace @ 4:10 to temp @ 4:30

set 1255, T_{sample} ~ 1270

2.53 1.286 0.360 0.47 5.38 / (6.00) ~ 89.7 %

8/23/88 ISO 29, pellet IV (second "good")

2.81 1.455 0.438 0.7283 3.86 ~64.3 %

8/26

2.8 1.283 0.384 0.4964 5.64 ~94

ISO 30, pellet V (edge chipping during isopressing) O.K.

2.94 1.456 0.457 0.761 3.86 ~64.4 % consistent

4:20 to temp @ 1267C

2.93(5) 1.283 0.4— 0.517 5.67 ~ 94.5 ~65 h

2A

3 good slices

The above understood and witnessed by _____ Date _____ and by _____ Date _____

8-31

0.04g 0.33mm dia pellet set on edge of polished 211 substrate which itself rests on a piece of 211 resting in a Al_2O_3 boat on a bed of 123. Adjacent to substrate is small pellet of 211 to allow eutectic pellet to straddle edge of substrate to minimize contact.

Heat treatment: 10°C/min to 1000C in flowing O_2

previous exps. in air/O_2 showed incongruent melting of eutectic @ ~1000C.

10:45 A.M. T@ 500C ∴ 1000C plateau should be reached @ 11:35

Will allow to melt for 1h → 12:35

10C/min → ~~785~~ 1:00
5C/min → 600 1:30 hold
~~10C~~ → ~~quench 2:30~~
→ 10C/min → 300C quench

Flow not pronounced. Not alot of liq. formation. Pix taken.

Redo in Air/O_2 where prev. exp. showed alot of liq. formation.

The above understood and witnessed by

Date

and

Date

9/6 $Bi_{2.15}Sr_{1.68}Ca_{1.17}Cu_2O_{8+\delta}$ (X-RAy single ϕ)

2 2 1 2

$Ca_{(2-x)}Sr_xCuO_x$ ← Nature letters $(1-X)$ $Ca_{0.86}Sr_{0.14}Cu_1O_2$

$Bi_2Sr_2CuO_x$ 2.1 1.6 & 2.2 1.6 so use 2.15 1.6 1.0

Bi	208.980	Bi_2O_3	~~465.9582~~ ✓	g/M (2 moles!)
Sr	87.62	SrO	103.6194	
Ca	40.08	CaO	56.0794	
Cu	63.54	CuO	79.5394	

2.15 mmoles	Bi_2O_3	1.00181013 g	~~x 150~~ ⇒ 2.2	150.2715195 (2 moles)
1.68 ↓	SrO	0.17408059 mg	2.58	26.1120885
1.17	CaO	0.06561290 mg		9.841935
2.0	CuO	0.15907880 mg		23.86182
				210.087363 g

~~Sr~~CO_3 - 60.00935
$SrCO_3$ - 147.62935 $CaCO_3$ - 100.08935

$\frac{SrCO_3}{SrO} : \frac{147.62935}{103.6194} = 1.42472693\ (26.1120885) = 37.20259576$

$\frac{CaCO_3}{CaO} : \frac{100.08935}{56.0794} = 1.78477926\ (9.841935) = 17.56568146$

The above understood and witnessed by _____ Date _____ and by _____ Date _____

22g/6

① Mixed, ground and calcined @ 775C 21 h (PE)

② grd and kept in Al_2O_3 boat covered w/ Au foil @ 800C for 6 h

③ grd and recalcined @ 850C for 16h

④ grd & mill for pelletization

factor of 2 molar correction

150.2715195 / 4 = 37.56787988 → 18.78393994 g

37.20259576 — 9.30064894 (0.7018889) = 6.528

17.56568146 — 4.39142037 (0.5602933) = 2.4605

23.86182 — 5.965455

57.22540 ~~(0.7018889)~~
− 1.931
− 2.7726
52.522 g batch

scale up everything by 1.5

28.176
13.950 ⊛
6.587 ⊛
8.948
57.661 (less CO_2 loss*)

13.95 (.7475) = 9.7895
6.59 (0.560) = 3.69

4.16 g loss
2.9 g loss
7.06 total expected

9-7

	calc'd	weigh'd	GRAVREC
Bi_2O_3	28.176	28.18	207.28 ← bottle tare; post 207.30(1)
			235.45
			28.17 //
			Δ + 0.03
			0.03/57.7 < 0.05% loss
SrO	13.950	13.96	235.45
			249.41
			13.96 //
CaO	6.587	6.59	249.41 (2)
			256.02
			6.61 + 0.01(2)!
			−0.01(2) recovery better
			6.60
CuO	8.948	8.96	256.00 (0.02)
			264.96
			8.96 ✓

① Pwdr. transferred to 0.D. tall bottle, shaken for > 15 mins.
② ↓ to 400ml beaker w/ ~150ml (made up to) ~~400~~ 150
③ Continuously stirred w/ mgg. stirrer while removing solvent - 11:30 → 1:20
④ Stirrer removed lowered to 'low', dry for overn @ 2:00
In oven under vac @ "3" 2:05 → 3:45

146.59 (8) beaker 159.46
89.12
57.47 vs 57.69 (0.4% loss) ~~may be some~~

152.23 (2) w/ top

146.59
142.15 post 20 NO EVIDENCE OF VAPOR
4.44 g loss ⇒ 7.06 g expected ∴ 63% conversion if no Br loss
89.12
53.03 ~51.26 52.86 mc 99.7 % > Post grd 51.43 97%

24 9/9

BiSrCaCu Calcination II : gold lined Al_2O_3 boat

86.51 barely fits in larger boat
35.13
51.38 vs 51.43 0.1% transfer loss

85.10
35.13
49.97

49.34 IN furnace (tube) for 850C, 16h calcination

84.50(48)
.21 stuck in screen
84.71

83.58
35.13
48.45 need on X-RAY

$Bi_{2.15}\ Sr_{1.6}\ Cu_1\ O_x$ (ref. data pg 21)

2.15 mmoles Bi_2O_3 1.00181013 but $2Bi = 1Bi_2O_2$ ∴

Bi 0.50090507 mg/mM

1.6 mmoles $SrO \rightarrow (1.6)(103.6194) = 0.16579104$ mg/mM

1.0 mM $CuO \rightarrow$ = 0.0795394

$SrO \rightarrow SrCO_3 \rightarrow 1.42472693\ (0.16579104) = 0.2362(0696)$

scale factor for 50g lot ~60

(60) (0.50090507) = 30.0543 Bi ox

(0.23620696) = 14.1724 Sr as $SrCO_3$

(0.0795394) = 4.7724 / 48.9991 // Cu ox ~49g close enough

10-17-88 MIXTAKE ← NOT Applied

$Ca_{0.86}\ Sr_{0.14}\ Cu_1\ O_2$

0.86 (56.0794) = 0.04822828 4 (1.785) = 0.0861

0.14 (103.6194) = 0.01450671 6 (1.42472693) = 0.0206681 1

1.0 (79.5394) = 0.0795394

scale factor for 50g batch (340)

340 (0.04822828 4) = 16.398

(0.02068811) = ~~4.952~~ 7.027 (4.949)

(0.0795394) = 27.043

50.468 g (less O_2)

2.078

48.39

29.27
7.028
27.04
63.34

The above understood and witnessed by _____ Date _____ and by _____ Date

$SrCO_3$ decomp. @ 1340°C
Bi_2O_3 melt @ 880°C
$CaCO_3$ decomp 825°C
} CuO - 1026°C decomp, all below initial calcination 775°C

9/3 From Chandra:

$Bi_{2.15}Sr_{1.6}Cu_1O_x$ procedure for calcination - all Pt.

752C for 6h ← Grd. ✓
790 C overnite (16h)
~~(850C)~~
825C 16h NOT CONVERTED
890C < 855 20h

The above understood and witnessed by _____ Date _____ and by _____ Date

3
14/23 800C
223
11501 825C 27

9/8

① $Bi_{2.15}Sr_{1.6}Cu_1O_x$

	calc'd	x'ed'd	
Bi_2O_3	30.0543	30.06	201.50 tare 231.55 30.05 ✓
$SrCO_3$	14.1724	14.18	245.72 14.17 ✓ (9.98 oxide)
Cu_2O	4.7724 .999 = 4.777 ↗	4.78	250.40 (49) 4.78 ~ ✓ OK.

$Ca_{0.86}Sr_{0.14}Cu_1O_x$ ←

$BaCO_3$	16.398	16.40	211.32 (3) tare 227.74 (2-6) ③ 16.42 recovery } slightly unstable 239.79 7.03 OK
$SrCO_3$	2.027	7.03(4)	
Cu_2O	27.043	27.04	261.81 27.04 ✓ .03 added 27.07
	27.043/.999 = 27.07		

Shaken, suspended in ISO, mixed & dried as per 2212 previously.
Will start both tomorrow after consultation w/ R. Fyat.
Merrill says try 800C to start w Pt.

No - transferred to HCl cleaned Bi SrCaCu crucible & fired for 6 h overnite.

137.87
89.15 tare (89.12) 0.03 exc.
48.72 / 49. expected 0.28/49 =
135.86 Post 6 ⇒ Greenish yellow (46.71)
−Δ2.01 (/4.19 expected) 48% reacted

135.51 (46.34)

133.28
133.66

Calcination II (post grind #1)

BiSrCu

135.51
89.15
46.34 -Δ 0.37 / 46.71 (0.8% loss)

133.66 post 790C 16h brownish-black hue, little sintering
89.15
44.51

44.10
132.66 let stand overnite, will run x-ray in morning.
post 825C 16h rich black texture, little sintering
exterior of sinter mass (tan?)

cont pg 30 →

Calcination II SrCaCu ~~to pg 33~~

140.92
90.48
50.43 / 50.52 0.03% loss

16.42 (0.56) = 9.1952 (-Δ 7.225)
7.03 (0.702) = 4.935 (-Δ 2.095)
-9.32

50.52
-9.32
41.20 ∴ 42.2/42.8 96%

133.28
90.49
42.79

42.43 post grind 0.8% loss. {Product looked good, little sintering, black.}

132.90
90.49
42.41

In furnace for 16h @ 1000C. Partial melting.
9/13 Run aborted. Restart. Cal I @ 800C then x-ray.

9/13 $Ca_{0.85}Sr_{0.14}Cu_1O_x$ RESTART // melting @ 1,130 w ant. 1,000 IN experimental synthesis

XSTALS excited Chandra. Same formula over.

9/14

	calc'd	exp'd
$CaCO_3$	16.398	16.41
$SrCO_3$	7.027	7.04
CuO	27.07	27.07

227.76
211.35 (4) tare
16.41

234.80
7.04

261.84 (5)
27.04 (5) ✓

9/15 RUN shot due to bumping when some got fanned up heat

#3

$CaCO_3$ 16.41

2
211.45
227.85 (6)
16.40 (1)

$SrCO_3$ 7.04

227.86
234.90
7.04 ✓

CuO 27.07

261.96 (7)
27.06 ✓✓

#4 - 9/21/88

$CaCO_3$ 16.41

211.49 (±1)
227.89
16.40

$SrCO_3$ 7.04

234.92 (±1)
227.89
7.03 ✓

CuO

261.98 (9)
27.06 ✓

The last time 9/21

$CaCO_3$	16.41	227.88 (9) 211.48 ~~211 TARE~~ (+1) 16.40
$SrCO$	7.04	~~234.93 (3)~~ ~~227.89~~ 7.04
C_oC	27.07	262.— (61.99) 234.93 27.06

Cold for Dick
22.87 g rec'd
8.92 owed
13.95 remaining

8.92
~~5.88 ot used~~
3.04 returned unused

VACATION ⇒ 9/26, ~~25~~ 24, 23, 22/88

After drying (n days) weekend

138.94
89.15 (14)
49.79 g recovered / 50.5 0.71/50.5 1.4% (bumping, etc)

VACATION 9/28, 29, 30/88

POST I 138.94
131.15
7.79 loss

131.15
89.15
42.00 g net

II finish
40.3 g collected after grinding for possible cal III

TO PAGE 32

PRE II
131.07
89.17

POST II 129.66
089.16
40.00

40.52
9.79 g loss

$B_{L2.10}$ $B_{L2.1}$ $B_{L2.2}$

9/14

43 gr $Bi_{2.15}Sr_{1.6}CuO_x$ collected after 16h 825C cal. } <100 mesh gr
will await x-ray tomorrow morning
Set-up jet mill. Dick still busy ck. X-ray. 9/19

10/3

Definitely NOT CONVERTED! X-RAY RUN.

10/4

Calcination II - 850C 20h

RE 132.19 − 89.12 = 43.01 Post 132.03 − 89.12 36h total: significant sintering has occurred probably "vent". MINOR sticking to crucible (Pt).

38.93 recovery

10/6

Cal III 875C 16h Sintered. Not really hard but has "metallic" luster. No melting.

(10/7) FINAL cal. X-RAY shows some small additional peaks, but predominantly "2201"

The above understood and witnessed by _____ Date _____ and _____ Date _____

10/4 900C melt test on partially reacted CaSrCu: nominally "0011"

NOTE: after 875 16h pattern discernable in x-ray but multicomponent MIXTURE. ABOVE TEST TO see if 900C cal safer.

8
126.92
86.66 tare
40.26

890C seems to be temp (+5C)

10/7 Chandra says 880C may be onset. Final cal. temp suggested as 865C

10/6 925C 16h: X-Ray shows

10/7 965C ~~16~~ h

8
126.37
86.66
39.71

appears as usual, some minor sintering. Still reacting, pattern loosing other peaks and increasing intensities of 0011.

966C ~60h

Switch to 810 when 812 went wild. One overshoot to 1006C for < 1 minute. Doubt if sample saw it. Removes some Pt exp on 1 side, harder than usual, reground & replaced for weekend stay. Hope for the best.

P_b-6 t_i=30 t_d=10 } optimization parameters (temp.)

The above understood and witnessed by ____ Date ____ and ____ Date ____

9/15

14.04
4.06

Skeleopycnometer density 2212: 6.45 g/cc

	g	t_{mm}	diamm				
(I)	0.21(5)	.211	.528	0.0462	4.65	72%	too high if correct 875C overnite melted/vaporized
(II) PRE Ref	(0.27)	(0.283)	(0.518)	0.0599	4.51	69.9%	lower
	0.265	0.293	0.574	0.076	3.49!	54	No way. (70 h) 852-5

yes! (11-3-88)

Sintering Temps - rapid temp

T_{set}	T_{cont}	T_{sample}		
830	836	858		
830	835	850	equil.	no sintering appreciable
855	863	875	equil.	incongruent melting/vaporization
835	—	855		
		851	equil.	no melting, looks pretty good

10/3 Analytical Results (ICP) 82.45 to 80

	wt %	mol %	(theor) MIX mol%	$+\Delta$mol%
Bi	49.0	2.34	2.15	6
Sr	15.8	1.78	1.68	3
Ca	4.85	1.21	1.17	✓
Cu	12.8	2.0	2	—

	m.w.	wt%-mol% conv.
Bi	208.98	8.28
Sr	87.62	1.38
Ca	40.08	0.631
Cu	63.54	1

UNNECESSARY

13.55 theoretical wt% ox @ 2212

82.45
~96.00

The above understood and witnessed by

Date

and

Date

 P2 - ~4500 150-28,500 **IBM Technical Notebook**

10/4

1.17 ~~0~~.095 0.278 0.262 4.465 69%

START SINTERING @ 4:00 PM Rapid temp setting 835. Should give pellet sintering temp of 855.

4:20 840 Tc promp ⇒ Ts 854 } slightly lower
846 Tc Read } overshoot

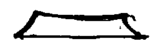
4:30 839 was Ts 859 Reduced
836

4:50 ↓ Stable @ 856

10/5

8:30 AM 16h sinter check

1.14 1.15(*) 0.287 0.291 (3.92)



Pellet warped, flowed ((*) approx. dia. due to non-vertical sides) and possibly has second ø RXN on part of top surface. Peak temp as far as I saw was 859. MUST keep below 850 (or at). C.

PRE

0.94 1.12 ~0.21 0.21 4.48 69.5 ~OK

pellet looks good @ 852 after 20 h (overnite) Keep sintering for grain growth

10/10

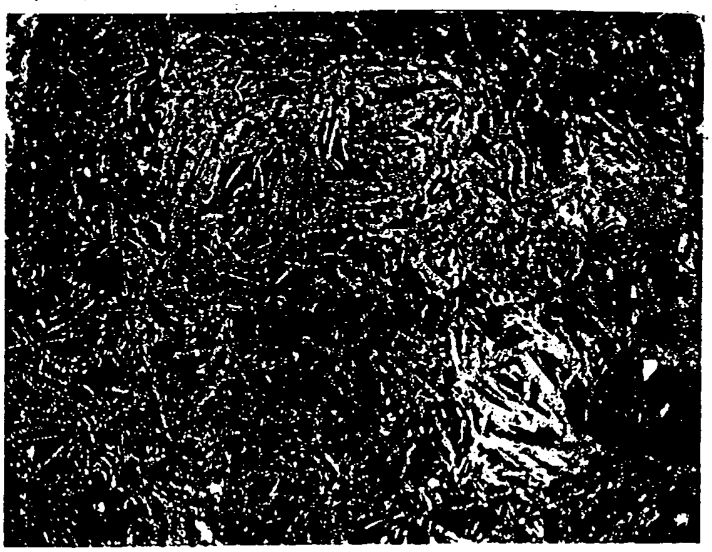
0.90 1.23 ~0.23 0.27 3.3 51% does not make sense

NOTE: T. SHAW SAYS people have seen such effects → same as previous results though

$Bi_{2.15}Sr_{1.68}Ca_{1.17}Cu_2O_{8+\delta}$

"22.12"

Analysis Results Pg. 33



1000X
120h
850C

The above understood

Date

10/6 2212 DYNAMIC SINTERING EXPERIMENTS

Sintering conditions: 850-854°C in Air/O_2 ; will need to preheat furnace to achieve short duration sintering times.

5 (15) (30) ~~45~~, ~~60~~ minutes @ first

10/13 Pre
1.29 1.093 0.311 0.292 4.42 68.5% UNL 3,750 ISO 27,500

In furnace 10:26 ~750 T_{stage} 11:04 out (38 min dill)
10:30 840
10:33 851 Start T_Δ sinter count (855-852)

Post
½ h 1.27 1.142 0.326 0.33 3.85 ~~60%~~ → 59.7

15 min Pre
1.19 1.095 ~0.288 0.27 4.41 68.4

In furnace 12:52 ~800 T_{stage} (plate cool)
(20 min dill) 12:56 850+ (prompting by/ upto T_{set} 865)
~~12:56~~ 850 ~ 852/3 1:12
1:01 854 Δ15 ⇒ ~ ~~[illegible]~~

Post
1.17 1.131 ~0.296 0.297 3.74 ~~61%~~ 61.1

Pre ~(1.1)
5 min 1.13 1.098 0.27 0.257 4.4 68.2
Post
1.11 ~1.108 0.271 0.261 4.25 65.9

2:32 → IN
2:34 → START } 853 ± 2
2:39 → OUT

The above understood and witnessed by _____ Date _____ and by _____ Date _____

36
10-9-13

1.2 g irregular pellet of powder "0011" isopressed to 27,500 psi and sintered for microstructural investigation @ ~972C.
Powder does not pelletize well @ all.

~2.h @ 972C 2ϕ

Post "2201"

1.21g 1.045 0.233 ~0.2 6.05 2hs ~~8.75~~ (peak 880C)

6.05/7.20 = 84%

NO PRE DATA

Post polishing data recheck

1.04 ~1.046 0.200 0.172 ~~6.05~~ 2hs ✓

10-17

0011 Synthesis

	theor.	weigh's	
$SrCO_3$	7.027	7.04	if 0.88 / 6.03 / 29.95 (Δ = 0.68)
$CaCO_3$	29.27	29.28	
CuO	27.04	27.05(4)	

$CaCO_3$ 240.77 − tare 211.48 = 29.29 ✓

CuO 267.80 − 240.77 = 27.03 ✓

$SrCl_3$ 274.85 − 267.80 = 7.05 ✓

after mixing & drying: 63.15 / 63.37 = ~0.3% loss

PRE CALI
149.62(1)
tare 86.46(7)
63.15 ✓

Post 16h 875C
134.56(7)
86.46(7)
48.10

29.29 (0.5603)^{x} = 16.41 (x pg 21)
7.05 (0.7019)^{x} = 4.95
27.03 (1) = 27.03
48.39 total

w/ top 188.03 / 38.42 (Add to audit)

O_2 conv.: 48.1/48.39 looks complete

PRE CAL II (47.84)/48.1 = 0.5% grinding loss (to temp (966 @ 4:00 p.m. 10-19-88))

134.47
86.63
47.84

The above understood and witnessed by _____ Date _____ and by _____ Date _____

10-19-88
38

Note: 810 optimized process parameters

$P_5 = 4$ $t_i = 15$ $t_d = 5$ $\Delta P = 2.0$

Bi Synthesis Series #1

Variances

	2201	0011	2212
Bi	+3%	—	+8.84%
Sr	-6.25%	-7.1%	+5.95%
Ca	—	-4.5%⊗	+2.6%
Cu	✓	✓	✓

⊗ estimated: 0.82/0.86 a 4% (1.7%) t: corrected to Ca

	Anal / Theor		
Bi	2.22/2.15	—/—	2.34/2.15
Sr	1.5/1.6	0.13/0.14	1.78/1.68
Ca	—/—	0.82/0.86	1.2/1.17
Cu	1	1	2

Analytical Results

	2201		0011	
	~~Exact~~	theoretical	est.	theor.
Bi	2.21(5)	2.15	—	—
Sr	1.5	1.6	0.13	0.14
Ca	—	—	0.86⊗	0.86
Cu	1	1 ✓	1	

⊗ Note: Ca concentration off due to omission of oxide to carbonate conversion factor in initial calculations. Thus is consistent with large amount of CuO second ph.

2212		
Bi	2.34	2.15
Sr	1.78	1.68
Ca	1.2	1.17
Cu	2.0	2 ✓

10-26 0011 milled (10/25), X-ray indicates ~ single ϕ

die body : 0.483" / 1.228 mm I.D.

@ 8,500 psi pyde press too fragile to go in iso. left after a few attempts @ passing w/ resultant crumbling.

Next time : ~ 3,600 psi ⇒ 16,000 may need to remill, PSD not available presently.

Pre data not taken;

POST : 875C for 3h (peak-5mw-@886C) Rapid temp find at 948

1.36+ (pellet damage ⇒ ~1.4) 3.68/4.86 = 75.6 ⇒ 76. (damage)

1.174 0.352 0.381 cc 3.675 NEED pycnometer density.

Sintered microstructure reveals ~ 80-86% dense pellet w/ minor 1-2% probably CuO phase in some triple points. ~~Grains ... color wobbling (light / dark yellow) ⇒ crap film~~

1000X, 3h sinter, 0011, POLARIZED

The above understood and witnessed by _____ Date _____ and by _____ Date

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY 4.95 theoretical

Density - 4.87

SAMPLE I.D. Comp: 0011" DATE 10-27-88
SOURCE SW JT OPERATOR PRD
TOTAL WEIGHT 19.02 g. OUTGASSING CONDITIONS Ar
TARE WEIGHT 4.06 g.
SAMPLE WEIGHT 14.96 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

3.685 3.695 (DATA) 3.691 3.692

	RUN 1	RUN 2	RUN 3	RUN 4
P_2	19.646	19.831	19.683	19.718
P_3	5.331	5.392	5.333	5.341
V_p	2.999 cc	3.076 cc	3.07 cc	
DENSITY	4.99 g/cc	4.86 g/cc	4.87 g/cc	

III. DENSITY WORKSHEET

STEREOPYCNOMETER
TRUE POWDER DENSITY

SAMPLE I.D. 2201 DATE 10-27-88
SOURCE OPERATOR PRD
TOTAL WEIGHT 19.04 g. OUTGASSING CONDITIONS N_2
TARE WEIGHT 4.06 g.
SAMPLE WEIGHT 10.98 g. ADDED VOLUME, V_A 85.52 cc
CELL HOLDER VOLUME, V_c 34.85 cc

OPERATIONAL EQUATION $V_p = V_c + \left[\frac{V_A}{1 - P_2/P_3}\right]$

V_p = Volume of Powder (cc)
V_c = Volume of Sample Cell Holder (cc)
V_A = Added Volume
P_2 = Pressure Reading after Pressurizing Cell
P_3 = Pressure Reading after Added V_A

3.568 3.566 (DATA) 3.566 3.566

	RUN 1	RUN 2	RUN 3	
P_2	19.865	19.720	19.807	19.661
P_3	5.568	5.530	5.555	5.514
V_p	1.548 cc	1.522 cc	1.522 cc	1.522
DENSITY	g/cc	7.21 g/cc	7.21 g/cc	

0011 Rapid Temp Sintering Process parameters

10-31

T_{set}	T_{sample} (equil. values)
962	986
950	978
948	977

note: preheated chamber and overshoot Tset needed w/ back-off on approach to get quick sintering temperature attainment

The above understood Date and Date

Diffusion Pellet Calculations: DOPSD!

123 std. pellet volume: 3.25g/6.36g/cc = 0.51 cc

∴ 2201 = 0.51 cc x 7.2 g/cc = 3.67 g ~3.75 g

0011 = 0.51 cc x 4.86 g/cc = 2.48 ~2.5 g

In preheated RT @ 3:31

	T_{set}	T_s	T_{sample}		
0011	948	9??	933	3:31	
		956	965	3:33	
	958	964	974	3:35	backing off now to maintain temp. till EQ
	950	956	974	4:20	
	951			4:21	for nite

11-7-88

IBM Technical Notebook

Sovey 2212 sintering time versus rel density

h	Rel D(%)	wt
0	68.4	
0.08	65.9	0.97
0.25	61.1	
0.5	59.7	
(16)		
70	54	small pellet, density determination probably not as accurate
120	51	

#3 16 h (not listed in book)

POST

0.88 1.15 0.216 0.22 4.0 / 4.45 62

REDO

10-2-88 0011 Analytical

Sample Nomenclature 43

El.	Wt %	theo. M%	Anal M%
Ca	22.4	0.86	0.875
Sr	8.24	0.14	0.147
Cu	40.6	1	1

Example calc.
Ca wt% / Ca mol Wt = 0.639 / .634 = 1
Sr wt% / Sr mol Wt = 0.094 / .639 = 0.147
Ca wt% / Ca mol Wt = 0.559 / = 0.825

11/3
0011 pellet 2 for 16h diffusion sinter
PRE 4,000/30,000 slightly irregular

2.85	1.531	0.496	0.913	~3.12	/4.95 = 63— perfect
2.81	~1.36	0.44	0.639 / 0.64	4.4	= 89

8201 4,000/30,000 PRE

3.78	1.365	0.494	0.723	5.23 = 72.4 % (too high?)

11/4
0011-2 cut into 2 slices. Didn't add block thickness so unequal.
1 ~ .230 cm thick 1 ~ 0.179 cm
will use for first press

Post
875C for 30 hrs ⇒ pellet has warped, grown large rodlike xstals and sagged. Obviously metastable.

previous 3h sinter showed no evidence of instability.

~~11-28-88~~

The above understood and witnessed by _____ Date _____ and by _____ Date _____

11-9 2201 pellets

IBM Technical Notebook

3200/28

201-3	2.99	1.351	0.415	0.595	5.02	69.7	open porosity
	2.96	~1.306	0.372 (→0.37)	0.498	~5.98	~83.3+ (defects)	pycnometer 86 ave = 85

2-3 MIN 872C SINTER (SLO ATTAINMENT 12 MIN VERSUS 1 ∴ $T_{SINT} = T_{AT} + 3$ MIN)

3300/30

201-4	1.16	1.082	0.245	0.225	5.16	71.7	5 min.
	1.15	1.050 */ 1.10 †/		0.182	6.32	87.5	double-S

201-5	1.17	1.086	0.248	0.230	5.09	70.7	broken before sintering

reground, repressed pellet 201-11

201-6	0.99	1.096	0.205	0.193	5.13	71.25	to temp 1845 out 2:15 30 min
*†	0.98	1.06	0.185 (1 lip)	0.163	6.01	83.5	
*			~(0.178)	0.158	6.2	86	

*(†): 201-4,6 some evidence of drooping 

. reduce temp 5C

2700/29

201-7	1.2	1.086	0.258	0.238(5)	5.03	69.9	to temp 2:27-8 15 2:45 15
1.01 1.016	1.19	1.05	~0.225	0.195	6.1	84.7	

The above understood and witnessed by

Date

and

Date

11-10 Sintering Summary <2201 data>

201-2

	temp	SINTER T	green	POST
201-8	872	2 min	71.25	85.6 ← 011-2201 P3 pressed pellet
201-3	872	2-3	69.7+	86 ← (large die)
201-4	875C+	5	71.7	87.5
201-9	872C	5	71—	84.7
201-7	872C	15	69.9	84.7
201-6	875C+	30	71.25	85.5
201-10	872C	1h	70.4	~~86 ?~~ → 85
201-1	875+	2h		84—
201-2	875	30h		~~(76.1)~~ → 79

86.1 ave

Record keeping: 201-2 30h 875C ~75% (pyc): IRregular pellet growth resulting in varying local densities
201-3 0.608 dia. pellet for pressure diffusion sinter
201-5 regrid → 201-11

11-11 GAS pycnometry gives an averaged rel density for pellets 1, 7, 8, 9 (wgt 4.6g) of 86.75g vs 84.75 (reasonable agreement), mostly closed porosity.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

46

11-14 SINTER 870-875C NOTE: [ALL SINTER TIMES ARE 1 MW attainment + 1 MW EQ SOAK + Δ SINTER time]

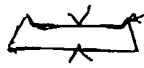
201-8 perfect pellet ~3000/29000

	1.19	1.081	0.253	0.232	5.13	71.25	green
post 2 min	1.17	1.038	0.225	0.190	6.16	85.6	
201-9	1.2	1.075	0.259	0.235	5.11	71.00	green
post 5 min	1.19	1.036	0.231	0.195	6.10	84.7	
201-10	1.10	1.083	0.236	0.217	5.07	70.4	
post 1h	1.09	~1.057* (1.043-1.07)	~0.2†	0.175(5)	6.21	~~82.25~~ (accurate?)	see below

201-11 Pellet was regrd & repressed from broken pellet. Also, die RAM froze resulting in much higher uniaxial pressure (of true rdg.) 12,000

	1.169	~~0.252~~	0.21	5.04	70.08

* linear average dia due to slumping. (- 

† W pellet interior after edge ridge worn away 

? probably slightly less due to exclusion of edge volume and linear ave approx

after flattening; 15 μm

201-10

	0.90†	~1.057	0.168	0.147	6.12	85 better (more accurate)

11 2.96 (2.98)

201-11 1.357 0.412 0.596 4.[illegible] ~~4.96~~ 5.0 (69.4)

2.935 ~1.315 0.365 0.496 5.94 82.5

2.945

201-11 cut "in half" longer flattened and polished.

0011-2201 sandwich ~0.353-0.363 thick.

> From furnace top to bottom of "weight plate" 1 9/32" @ 462C

assuming ~6 lbs for ram & plate & x-sectional pellet area of 0.212 in^2 load ⟹ 28 psi

Diffusion sintering set @ 860C for ~12 hrs.

Rel density from measurement of 201-11 ~83 %. On inspection of internal polished surface numerous burnout-like occlusions present. Some degree of open porosity, also.

Pyc. rel. den = 88. % thus Δ attributable to open porosity.

0011 Rel density from measure ~ 89 %. No pyc reading done 16h sinter @ 975C.

4:30 pm. T @ 859C assume start of diffusion sintering.

Plate height 1 3/8" (3/32" expansion due to TCE from 462C & No RT measure made, but not significant)

The above understood and witnessed by _____ Date _____ and by _____ Date _____

11-28-88 <INSERT>

Results (by microprobe) of $CaSrCuO_x$ melt xstals

Melt composition was from pgs. 27-29

Composition was <u>not</u> $Ca_{0.86}Sr_{0.14}Cu_1O_x$ in melt, but rather

→ $Ca_{0.48}Sr_{0.14}Cu_1O_x$ ←

from which xstals grew of $CaSrCuO_x$ with stoic.

$Ca_{0.81}Sr_{0.2}Cu_1O_x$ $x = 2.1$

Atomic wgt fractions were:

Ca	0.195
Sr	0.05
Cu	0.242
O	0.513 (by difference)

Melt temp. was 1000C for 16h with cooling virtually but NOT TOTALLY A QUENCH: UNCONTROLLED RATE REGULATED BY FURNACE thermal MASS

11-22

Balance Bi Packs for Rxn

2212 - ~g 30.5

2201 - 12.5

0011 - 33.5

2nd Diffusion Run 2hr ramp to 866C @ 100C plate space = 1 7/32

0011 slice started @ 0.18 cm (not measured when finished, either was 2201 or sandwhich, will try to approx)

2201 ~ same 0.18

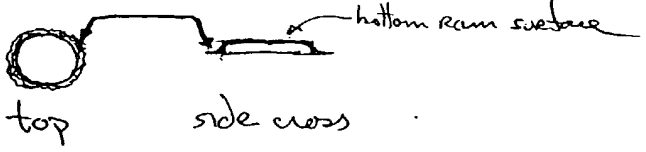
loose ~ 0.23 - 0.2 ≈ 0.03/slice ∴ 0.18 → 0.15 0.17 → 0.14

so sandwhich might be ~ 0.29 cm (80% of Run #1)

Approx thickness by height of plate differences @ 866C 1 12/32 - 1 3/32

3/32 = 0.23 cm ∴ .29 - 0.23 = 0.06 too small → 871C peak

RESULTS: "Bi" pellet has spread, apparently melting. Total thickness 0.18 cm. Liq generated crystalline (?) skirt around pellet periphery.



0.18 cm = 0.07" slice ~ square 0.07 + 0.015 = 0.085

0011 - 2201 Mix Calculations

wt% 0011	wt% 2201
~~x~~ ✓	+2
~~x~~ ✓	+5

$Bi_x Sr_z Ca_1 Cu_2 O_8$

$Bi_2 Sr_2 Ca_0 Cu_1 O_6$

$Bi_0^3 Sr_{"0"}^2 Ca_1^1 Cu_1 O_2$

From "ideal" stoic. 2201 + 0011 → 2212

	A.W.	0011	2201 (1 mole)	2212 (1 mole)
Bi	208.98	—	417.96	417.96
Sr	87.62	—	175.24	175.24
Ca	40.08	40.08	—	40.08
Cu	63.54	63.54	63.54	127.08
O	15.9994		95.9964	127.9952
		135.6188 +	752.7364 =	888.3552
Bi		0(0.14)(0.86)1 —	(2.15)(1.6)(0)(1) 449.307	(2.15)(1.68)(1.19)(2) 449.307
Sr		12.2668	140.192	147.2016
Ca		34.4488	—	40.3284
Cu		63.54	63.54	127.08
O		31.9988	~95.9964*	~127.9952
		142.2744	749.0354	891.912296 / (891.3098)
% dev		+8.6%		99.6% (2.15 etc)

99.9%

*(5.825)15.9994
93.1965

The above understood and witnessed by ... Date ... and ... Date

CONTINUATION...

1 mole "0011" + 1 mole 2201 → 011 + 529 wt% 2201

142.2744g 752.7364g

142.2744g + (0.02)(142.2744) = 145.12g] 2 wt% batch size
2.84549

factor 15
9.49
9.485 0.19 = 9.675

142.2744g + (0.05)(142.2744) = 149.38812g] 5 wt% batch size
7.11372

9.485 ~0.475 = 9.96
0.48

For Stoic (molar) mix = 1.423g + 7.527 = 8.95g batch size

Total Usage	0011	2201	
	20.393	8.192	
%	61	66	

	cc	cc	vol%	wt%
Vol table	2.37	0.0264	1.0%	2
	2.37	0.0736	3	5
	0.356	1.045	25%	stoic

STOIC:	Bi	Sr	Ca	Cu
"2201"	2.15	1.16	0	1
"0011"	0	0.14	0.86	1
	2.15	1.74	0.86	2

versus poly 2.15 1.68 1.17 2

The above understood and witnessed by Date and by Date

Stoic Mixing

0011 - 2201

~1.43 g ~7.53 MIX STARTING @ 3:00 P.M., 50mls isopropyl.
5cc ZrO_2 balls
2/3 full

NOTE: ~~From~~ bottom pg 51 can be seen this Additive approach will yield a theoretical molar comp { 0.1 M larger in Sr
0.31 M less in Ca

i.e. ~~Strontia rich, Calcia poor~~

8.96 g added initially, 8.85 g recovered ∴ 1.2% loss (98.8 yield)

Stoic 1 Pre 2700/27,500

3.11 1.36 0486 0.706 4.41 ~68?

0.25 (4) + 0.75 (7.2) = 6.4 vol% basis, ~ density calc

Rxn. (sinter) temp to be 850C

Pellet melted indicating lower mp liq ϕ exists in system (later xstallized). Predominantly 1 lath-like ϕ w/ exaggerated growth as in 2201 12th sample.

The above understood and witnessed by Date and Date

12-5

4:20 P.M. // 4:25 @ temp.

0011-3 placed in preheated Rapid temp set @ 951C (T_{sinter} = 975C) for overnite sintering.
No pre data on density due to irregular shape caused by pellet crumbling during Isopressing.
Unipress → 6000, ISO - 29,000 PSI wght ~ 3.1 g 3.0-2.9

12/6
9:30 Slow cooling began ∴ ΔT_{sinter} = 17h @ 875C

Post 2.86g ~0.460 mm thick radius might have been ~1.3

estimated density 0.666cc @ 3.1g ~ 4.6 / 5.00 ≅ 93 (may be high)
3.0 4.5 / ↓ 90 better

0011-3
0.181" thick
Slice 1 → 0.09" after cleaning // post polish → N/R
Slice 2 → 0.074
0.179

2201-8 1.038 dia ∴ area = $\pi D^2/4$ = 0.85 cc^2
0.409 = 0.525 in^2 5.75 lbs / .525 in^2 ~ 11 psi

Pellet configuration @ START ~ 3:55 p.m. thickness - 0.34cm

Ramp → 434 Set point - 800C Dwell - 12h 1 7/32 @ 380C

12/7 Result: no melting, pellets bonded w/ little deformation.

12/8 After 24h 825C anneal no evidence of liq., but bond breaks after handling at pellet interface with some "rxn etching" of 0011 pellet surface leaving thin uneven layer of 2201 (or rxn prod) behind.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

12-6

IBM Technical Notebook

~~Ref pg 25~~

SECOND 2201 Synthesis $Bi_{2.15}Sr_{1.6}CaCu_1O_6$

Bi as Bi_2O_3: 30.0543 x 2 = 60.1086 60.11

Sr as $SrCO_3$: 14.1724 28.3448 28.35

Cu as CuO: 4.7724 / 48.9991 g → 9.5448 / 97.9982 g 9.54

~ 0.7019 conversion factor for $CO_3 \rightarrow O$ 28.3448 (0.702) = 19.898

Estimate ~ ~~89~~ g "batch recovery" 97.9982 − 8.4468 CO_2 loss = 89.55

12-7

tare 202.54
Bi_2O_3 262.68
60.13 − 60.11 = +0.02 ✓

~~tare 262.~~

$SrCO_3$ 291.03 ← 291.[illegible]8.35 wgd 28.36 twofnd 28.36
tare 262.68
28.35 Δ ✓

CuO 300.57 (300.57/9.54) wgd 9.55
291.03
9.54 Δ ✓

12-8 97.92 / recovery after drying overnite
98.02 theoretical = 99.9% yield 0.1% mixing loss

to pg 56 →

The above understood and witnessed by Date and Date

12-7-88

0011		2201	
9.49		0.48	theor.
9.48		0.50	wigd
0.02		>.01	read
9.46		0.49	actual = 9.95

Std. 1.-1.5h 5min ZrO_2/Iso grind mix, screening & drying.

12-8-88

Recovery : 9.84g / 9.86g theoretical = 99.8% >0.2% loss

60.87
cont tare 51.04/5
9.83 transferred

0011-2201-5W(3V)-1
Post 8500/29,000
2.31 1.117 0.704 0.690 3.35 ~67%

Pellet bigger than usual, 1.75g max in future might be considered.

— 1.183 0.715 CRACKED; measurements N/A

12-9
5W-2 900C 8500/30,000

1.27 1.174 0.382 0414 3.07 61.4!

3:55 in preheated furnace → 4:00 to temp @ 900C
POST 5 MIN
1.24 1.111 0.36 0.349 3.55 71 —

15 MIN NO SIGNIFICANT CHANGE

12-12 to temp ~ 10:30 A.M. (check: 10:45 → no slumping) → SINTER till 12:30
12:15 A.M. slo-cool initiated
~~11:45~~.
~2h
1.24 1.055 0.33 0.29 4.28 ~86%

12-8-88 2201 SynII cont. (from pg 54)

crucible tare 186.68 ~~spill~~ → 186.69 → 192.29 w/ top
88.79
97.89

10:00 A.M → 575C hold 1h
11:00 → 800C
12-9 11:00 AM cool, required to < 100 next

182.23
88.95 (wgt after sintered parts body removed)
93.28

~~93.44~~ if 88.79 used
182.02 after grinding
88.95
92.07 to temp. (866 C.) @ 1:00 p.m.
-Δ 1.21 in grinding 1.3%
~~97.88~~ - 93.28 = 4.61 } 55% REACTED
97.89 - 89.55 = 8.34 }

1:00 - 5:00 pm 866C, shut down for weekend (Away resident scientist)

12-12-88

to temp 866C @ 10:00 A.M.
off @ 7:00 A.M. 12/13/88

PARTIAL MELTING, "classic" eutectic lamellar and large 2201 lathes.

The above understood and witnessed by Date and by Date

12-14-88 2201 SynIII

working jacket : 202.75

58 12-14-88 SynIII 2201 IBM Technical Notebook

$Sr/Bi = 0.744$

$Sr\ 0.8\ \ S_r = 1.72$

mixing jar tare 202.75

Bi as Bi_2O_3	30.0543	× 2 =	60.11
Sr as $SrCO_3$	14.1724		28.35
Cu as CuO	4.7724		9.55
			98.01
			− 8.5 CO_2 loss
			89.51

Bi_2O_3 262.86
tare 202.75
60.11
291.20 (19)
262.86
28.34 (5)
300.75
291.20
9.55

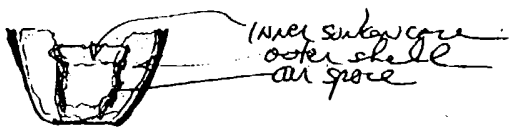
PRE CAL I

crucible + 185.84
tare 87.99
97.85 97.85/98.01 0.2% mix loss

12-20 Post 750C 16h calcination
crucible + 181.15 NO RXN w/Pt.; lime green cake/bottom, uniform throughout
87.98 except for top ½ edges (grey)
93.17 93.17 − 97.85 = −4.68 /8.5 = 55.% CO_2 lost
post grd 92.70 post gr

crucible + 180.70
87.98
(92.72)

12-21-88 Post 790C 20h calcination
178.10 Material looks very good, smooth, indicating lamilar structure
87.98 Uniformly black, sparkling sinter body; An outer shell
90.12 / 89 ≃ RXN close to completion @ 98+%
AND inner core structure, (see below)



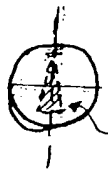
to page 60

The above understood and witnessed by _____ Date _____ and _____ Date

$Y_1Ba_2Cu_3O_x$ Implantation Experiment

PRE - film on $SrTiO_3$ 3500/30,000

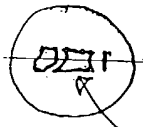
3.07 0.448 0.485 0.799 3.84 ~60.4 %



← line 'marker' ⊥ to long axis of triangular $SrTiO_3$ implant
implant orientation – NOTE: MARK ON UNDERSIDE of pellet
∴ film side opposite

3.02 1.271 0.396 0.502 6.02 94-95

3.05 1.498 0.476 0.784 3.89 ~61



← line 'marker' ∥ to long cutting axis of two pellets (cut on line
implant orientation – see NOTE above for polished side orient

2.99 1.272 0.391 0.497 6.02 94-95

5

5:12 p.m. 475C @ 10C/min to 975 Δ500C/10C/min = 50min ∴ 6:00pm

Cutting $SrTiO_3$ implant: measures 0.5" on saw (0.485–0.505 tangents)

1.222/2.54 = 0.481 ✓

The above understood and witnessed by _____ Date _____ and by _____ Date _____

12-21-88 Calcination III 2201-B3

X-RAY SHOWS DISTINCTLY NOT SINGLE ϕ, even though material looks "O.K."

total 177.04
cwx 87.98
89.06 88.2

- 87.98
+ 88.2
0.22 g GAIN

12-22-88

175.7— (-Δ 1.34) slight sticking (RXN) w/ cwc. bottom
87.98
87.72 - 89.51 (theo.) = 1.79 g greater than theoretical loss
could be grinding loss 2%

PRE 850 Cal

174.93 total
87.93 tare (after acid cleaning)
87.— g 0.72 g grinding loss (consistent w/ previous losses)
+ X-ray slide

POST 12-22-88

174.83
87.93
86.90 — -Δ 0.10 100% RXN ! NCONSTANT WEIGHT

NOT superconducting, but not surprising.
batch #1 wasn't either

Summary various RKN pellets:

5 wt% 2201 in 0011 for 16h @ 850C SEM

5 wt% 2201 in 0011 for 2h @ 975C SEM

[0011 - 2201 ~~850C~~ mixture ~~pressure bonded~~ pellet: 13h 850C
no Ø formation, exaggerated grain growth/warpage] later

0011 @ ~~975~~C 17h SEM STD.

2201 @ 875C 1h SEM STD.

2212 @ 853C 5 MIN SEM STD.

12-29-88 Dave's Compositions

#	Y	Ba	Cu	Y	Ba	Cu
	1 (0.167)	2 (0.33)	3 (0.50)	0.17	0.33	0.50
	0.15	0.33	0.52	0.8639	1.9038	3—
	0.17	0.35	0.48	1.0625	2.1875	3—
	0.19	0.33	0.48	1.1875	2.0625	3—
	0.19	0.31	0.50	1.14	1.86	3—

Calculated Compositions (calculations next page)

	Y	Ba*	Cu	total	
1)	1.91937 (1.92)	6.51253 (6.51)	3.97697 (3.98)	12.48	←
2)	1.69356 (1.69)	6.51253 (6.51)	4.13605 (4.14)	12.34	←
3)	1.92	6.90723 (6.91)	3.81789 (3.82)	12.65	
4)	2.14578 (2.15)	6.51 0.33	3.82 0.48	12.48	←
5)	2.15	6.1783 (6.18) .31	3.98	12.31	←

* Ba as $BaCO_3$
Y as Y_2O_3
Cu as CuO
} NOTE ⇒ no purity corrections applied yet

1/3/89

Calculations for wgts SUMMARIZED ON page 62

2) $Y_{0.15}\ Ba_{0.33}\ Cu_{0.52}$

$Y = 0.15\ (225.8082)/2 = 16.9356$ g Y_2O_3 1/3/89

$Ba = 0.33\ (197.3494) = 65.1253$ g $BaCO_3$

$Cu = 0.52\ (79.5394) = 41.3605$ g CuO

3) $Y_{0.17}\ Ba_{0.35}\ Cu_{0.48}$

$Y = 0.17\ (225.8082)/2 = 19.1937$ g Y_2O_3

$Ba = 0.35\ (\quad) = 69.0723$ g $BaCO_3$

$Cu = 0.48\ (\quad) = 38.1789$ g CuO

4) $Y_{0.19}\ Ba_{0.33}\ Cu_{0.48}$

$Y = 21.4518$ $Ba = 65.1253$ $Cu = 38.1789$

5) $Y_{0.19}\ Ba_{0.31}\ Cu_{0.50}$

$Y = 21.4518$ $Ba = 61.1783$ $Cu = 39.7697$

1) $Y_{0.17}\ Ba_{0.33}\ Cu_{0.50}$

19.1937 65.1253 39.7697

1/89

64

1) O_2 FIRED (TRIAL #1) → 88.5409 $Y_{1.17}Ba_{2.33}Cu_{3.5}$

Y_2O_3 - 1.92 CuO - 3.98(1) $BaCO_3$ - 6.51 6.5777 6.9665 6.9684

88.53 2.3140 4.3712 6.9665 6.9684

tare 76.06 0.3900 ✓ 0.3900 0.3888

12.47

1.922 3.98(2) 6.5777 = 12.48

post dry 12.43

↳ 99.6% recovery total - Δ = 0.05

1-9-89

10.95 g after 2nd 16h 950C O_2 calcination

$6.5777\left(\frac{153.3391}{197.3510}\right) = 5.11$ -Δ 1.47 12.43 1.47 10.96 ✓

0.777

1/17 D(AVE) post 1h grind = 1.86 μm 3000/30,000

P1 1.68 1.136 0.408 0.4135 4.06 63.8%

In 16h O_2 @ ~3:00 p.m. 1/10/89, to temp @ 950C projected @ 4:30 → 5:00 → 9:00

1.64 1.011 0.354 0.284 5.775 90.8 (91)

1/4/88

4) ~~Ox~~ fired O_2

$Y_{0.19}$ $Ba_{0.33}$ $Cu_{0.48}$

~~1.89~~ 6.51 ~~9.14~~ 3.82

~2.15

Y_2O_3 = 2.5342 6.9623 4.2083

0.3865 0.384 0.3865

2.1477 6.5783 3.8218 ⇒ 12.55 g

3.8217 · .3865 = 4.2082

2.1473 · .384 = 2.5347

1/10/88

Second ~~CALCINATION~~ STARTED after grinding. No evidence of liq formation. Pwdr looks good already.

10.99 g after 2nd calcination;

10.76 post grind

~~Yb~~ 6.58 (.727) = 5.11 Δ 1.47

12.55 + 1.47 = 11.08 g expected: 10.99 ✓ Recovered

1/17

P1 Pre 2500/30,000 to temp @ ~ 5:00 p.m.

1.60	1.14	0.399	0.4073	3.93	62%

1/18

1.58	1.055 v 0.365		0.319	4.95	77.8%

Green ϕ peaks coming up in X-ray.

The above understood and witnessed by _____ Date _____ and by _____ Date _____

5) $Y_{0.19}Ba_{0.31}Cu_{0.50}$

(.999) Y_2O_3 - 2.1473 (target 2.3760) $BaCO_3$: 6.1796 ① / .99 ⇒ 6.2407 (target 6.4686) CuO - 3.9810 ② (target 4.2092)

2.3761 / 0.2287 / 2.1474 ✓

tare 6.4690 / 0.2279 / 6.2411

4.2092 / 0.2282 / 3.9810 ⇒ 12.3695 ~12.37

12.34(3) collected after mix // 12.34/12.37 ~ Δ 0.24 % ✓

6.2411 − 4.8493 = 1.392

~~tare~~ 63.60 / 51.17 / 12.33

62.20 / 51.17 / 11.03

−Δ 1.3/1.39 = 93.5%

11.00 post grind

62.17 / 51.18 / 10.99

post CaII 62.11

12.63 / −.88 / 10.75

Post grind, 10.46

P1 9333/30,000 ⇐ PRE ⇐

P1	1.60	1.440	0.244	0.20	4.00	62.9
Post	1.56	1.266	0.210	0.26	6.00	(94 = 91)

Good densification, no apparent Ba, CuO islands present.

The above understood and witnessed by ____ Date ____ and by ____ Date ____

2201

P1B3 2500/30000
PRE
1.94 1.09 0.391 0.365 5.315 73.8%
Post 15 min @ 860
1.94 1.06 0.358 0.313 6.2 86%

2 wt% 2201 IN 0011 1/13/88

9.49
0.19
9.58

some leaking during 0.5h mix : 9.05 g

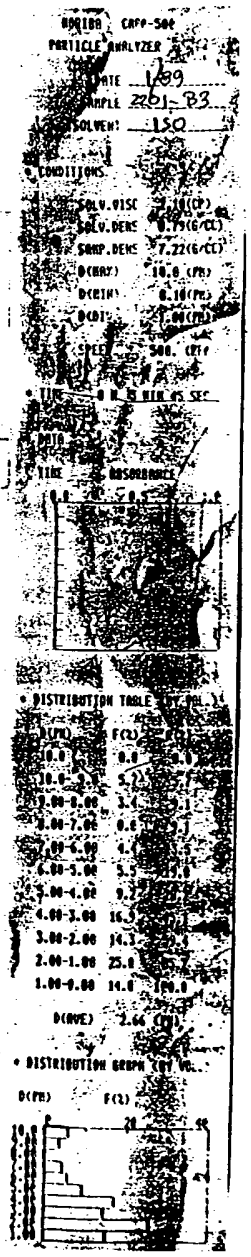
9.05 g
8.68 g final
0.37 g loss

8.68 g
0.37 g

2 at% P1 4200/38,000 to temp @ ~5:00 p.m.
Pre
1.70 1.163 0.499 0.53 3.21 ~64.2 (~~80.25%~~)
1.69 ~1.21 0.525 0.60 2.82 56.4
1.194 - 1.227
some slumping
-Δ 12%

Cuts yield : 1.57 mm inside ~~cts~~ 0.30
1.12 mm center 0.25
polished → 1.79 mm outside 0.180 (taw)

+	26.52	26.03	25.94
post	25.52	25.52	25.45
	1.00 →	1000 μms	490
		510	



2) $Y_{0.15}Ba_{0.33}Cu_{0.52}$ batch tare: 74.55

Y_2O_3 - 1.6936 $BaCO_3$ - 6.5783 CuO - 4.1361 .

(.2270'5) 1.9206 + 0.2270 ~ 1.6936 | 6.8066 + 0.2284(8) = 6.5782 | 4.3651 + 0.2288 = 4.1363 | total − (0.0004) ~ 12.41

1/17 1ST CALCINATION 66.53 (12.37 measured from mixing)
tare 54.16
12.37 ~/ 12.41 = -Δ 0.3%

6.5783 (.277) = 5.111 (-1.47) 12.37
− 1.47
~ 10.90 expected yield (less transfer losses)

1/18 post 66.53
65.15 (05)
-Δ 1.38 (48) tare 54.20 recovery 65.05
54.2
10.85 ~ total RXN.

2ND CAL (16h as above)

1/19 POST 65.02
54.2
10.82 ~ constant 10.99 Recovery

Notes: large liq. stains (formation) during 1ST/2ND cal unlike 1 & 4 where liq was suppressed in 1ST cal { minor in 2ND

PRE P1 3300/30,000 75C @ 5:16 ∴ temp @ 7:45, 16h ⇒ 11:45 A.M.

1.60	1.414	0.258	0.405	3.95	62.1%
1.57	1.227	0.216	0.255	6.16	96.9

1) O_2 4) O_2 2) O_2 3) O_2 1h iso PSD's 5) O_2

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-10-89
SAMPLE $Y_{0.7}Ba_{0.3}Cu_{0.5}$
SOLVENT ISO-1h

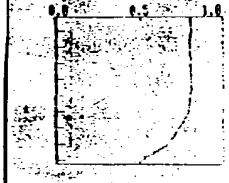
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE



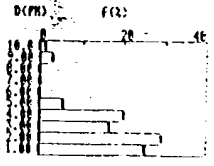
• DISTRIBUTION TABLE (BY VOL.)

D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 5.0	1.2	1.2
9.00-8.00	2.9	4.1
8.00-7.00	0.0	4.1
7.00-6.00	0.0	4.1
6.00-5.00	0.0	4.1
5.00-4.00	5.5	9.6
4.00-3.00	19.0	29.5
3.00-2.00	16.5	46.0
2.00-1.00	28.9	74.9
1.00-0.00	25.1	100.0

D(AVE) 1.8((PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-13-88
SAMPLE $Y_{0.9}Ba_{2.3}Cu_{4.8}$
SOLVENT ISO

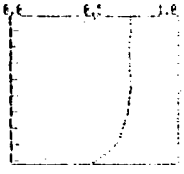
• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA 0.7

TIME ABSORBANCE



• DISTRIBUTION TABLE (BY VOL.)

D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 5.0	1.2	1.2
9.00-8.00	0.0	0.1
8.00-7.00	0.0	0.1
7.00-6.00	0.0	0.1
6.00-5.00	0.0	0.1
5.00-4.00	5.2	13.3
4.00-3.00	13.2	26.5
3.00-2.00	11.4	38.0
2.00-1.00	29.0	67.0
1.00-0.00	33.1	100.0

D(AVE) 1.66 (PH)

• DISTRIBUTION GRAPH (BY VOL.)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1/20/89
SAMPLE $Y_{0.5}Ba_{2.3}Cu_{4.2}$
SOLVENT ISO

• CONDITIONS D- 0.8

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

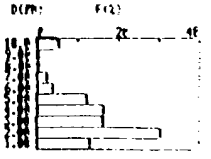
• DISTRIBUTION TABLE (BY VOL.)

D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	5.4	5.4
9.00-8.00	0.0	5.4
8.00-7.00	0.0	5.4
7.00-6.00	2.3	7.7
6.00-5.00	3.8	11.4
5.00-4.00	12.3	23.8
4.00-3.00	16.2	40.0
3.00-2.00	16.3	56.3
2.00-1.00	30.6	86.9
1.00-0.00	13.1	100.0

D(AVE) 2.39 (PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1/27/89
SAMPLE $Y_{0.7}Ba_{2.3}Cu_{4.8}$
SOLVENT ISO

• CONDITIONS SD- .96

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

• DISTRIBUTION TABLE (BY VOL.)

D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	1.0	1.0
8.00-7.00	3.5	4.5
7.00-6.00	4.1	8.6
6.00-5.00	6.4	15.0
5.00-4.00	7.7	22.7
4.00-3.00	11.8	34.5
3.00-2.00	14.3	48.8
2.00-1.00	20.8	69.6
1.00-0.00	23.2	92.8

D(AVE) 1.[illegible] (PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 1-24-89
SAMPLE $Y_{0.7}Ba_{0.3}Cu_{0.5}$
SOLVENT ISO

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	6.36(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 4 MIN 20 SEC

• DATA

TIME ABSORBANCE

• DISTRIBUTION TABLE (BY VOL.)

D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	0.0	0.0
9.00-8.00	0.0	0.0
8.00-7.00	0.0	0.0
7.00-6.00	0.0	0.0
6.00-5.00	0.0	0.0
5.00-4.00	0.0	0.0
4.00-3.00	0.0	0.0
3.00-2.00	0.0	0.0
2.00-1.00	21.6	21.6
1.00-0.00	26.6	48.2

D(AVE) 34.3 (PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)

The above understood and witnessed by ... Date ... and ... Date

	SrO	CuO
$Sr_7Cu_{12}O \rightarrow Sr_{0.37}Cu_{0.63}O_{19}$	38.34	50.1078
$SrCuO \rightarrow Sr_{0.5}Cu_{0.5}O_2$	51.8077	39.7697
$Sr_2CuO \rightarrow Sr_{0.67}Cu_{0.33}O_3$	69.4250	26.248

SrO = 103.6194 → $SrCO_3$ 147.63 14247

CuO = 79.5394

3.83 ~~5.02~~

3.834 5.0198 → 8.8538

(5.18) 5.18077 3.9697 (3.98) → 9.1579

(6.94) 6.9425 26.248 (2.63) → 9.5673

~~5.46~~	5.02	10.48
7.38	3.98	11.36
9.89	26.34	12.52
SrO	CuO	

CO11-5 at 1%201 @ 850C for attempted TEM prep.

slice 2 - 1.28 mm - 1280 μm

slice 3 - 0.68 680 μm

slice 2 prep: mounted side 1 measures ~ 27.69
26.30 - (33)
~ 1.34 - 1.29

1280
um: 300 μm 1340
980/2 = 490 = 490
850 target

~20 8's on "soft" 15 μm grits ~ 900 ; 150 8's on 6 give 770
720 μm before starting second side

26.40 after mount 39 → 26.21 150 → 26.16 150 6½ 26.06
25.69 25.69 ~4130 390 ✓
0.71 ✓ 520

The above understood and witnessed by Date and ... Date

3) O_2 fired $Y_{0.1}Ba_{0.35}Cu_{0.48}$

Y_2O_3: 1.9213 $BaCO_3$: 6.9770 CuO: 3.8217

2.1495 (2.1496) 7.2050 (7.2047) 4.0498 (4.0498)
0.2288 tare 0.2277 0.2281
1.9212 6.9773 3.8218
5.4214
1.55

Some bumping, but very good mix so should be fine. 12.35 post mix -Δ 2.3%

12.72 g expected RtDO (Bumping too critical)

Y_2O_3: 1.9213 $BaCO_3$: 6.9770 CuO: 3.8217

2.1425 (2.1496) 7.1973 (1.9770) 4.0425 (4.0427) total
0.2210 0.2200 0.2210
1.9215 6.9773 3.8218 12.72

Mix recovery after overnite drying: 12.63/12.72 -Δ 0.7% (acceptable)

~ 63.82
tare 51.19

Post 62.39
51.20
11.19

(11.17 expected)

CAL #I: 3:57 190C w5:15 est. temp attainment seems correct
4:25 486C ~ ✓

Post cal II

Post grind I: 11.21 → 11.14 62.35

62.25
51.20
11.05

Very little liq formation compared to #2. Apparently need excess Ba and Cu for large liq.

0.2 g loss due to Si contam from tube (pre-grind) 10.8

The above understood Date and Date

1/27

10.17 collected: white top layer on powder. Dry flake-cake very agglomerated/brittle and does not easily push out when brushed. Need to dry grind in order to produce decent push.

10:25 recovered after dry grind

P1 3500/29000

1.62	1.425	0.266	0.424	3.82	60—%
1.52	1.321	0.245	0.336	4.52	71 %

Pre grind & Post grind x-rays show change of some peaks in two x-rays, however sintering calcination of pellet may return rxn products to original ϕ's. Will do x-ray of pellet also.

Post: some slumping.

74, 2/16/87 12 Variation Study Property Pellets

#1 P2 3 rec/30,000 ave = 62.5

1.04	1.138	0.256	0.260	4	62.9
to 1.01	1.046	.22	0.189	5.34	84

#2 P2

1.02	1.123	0.264	0.261(5)	3.90	67.3
1.00	981	.223	0.168(6)	5.75	93.6

#5 P2 ✓

1.04	1.154	0.248	0.252	4.03	63.4
1.024	1.024	.224	0.184	5.54	87

Pellets in furnace @ ~10:30 a.m. 2/17/87 10°/min (pre-warmed)

t	T_c	T_s	
10:30	259	239	(?)
⇒ 10:45	442	516	+Δ 75°C
11:50	900±5	944	

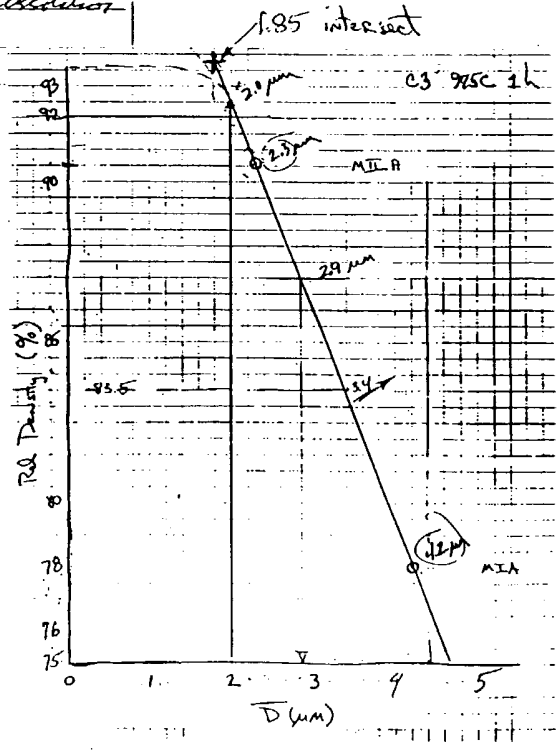
from early results Δ@ 30 went down by 25°C so estimated time to reach set point would be 45 mins. or 11:30 @ +Δ50 equilibrat at 950°C then relaxation over ensuing 20 mins of 6°C. Relax in platinum run equilibrate time was 13C as probably close.

1:45 pm 2/17/87 in furnace @ 600C project out 72 hrs.) 1:45 Mon.

See page A15

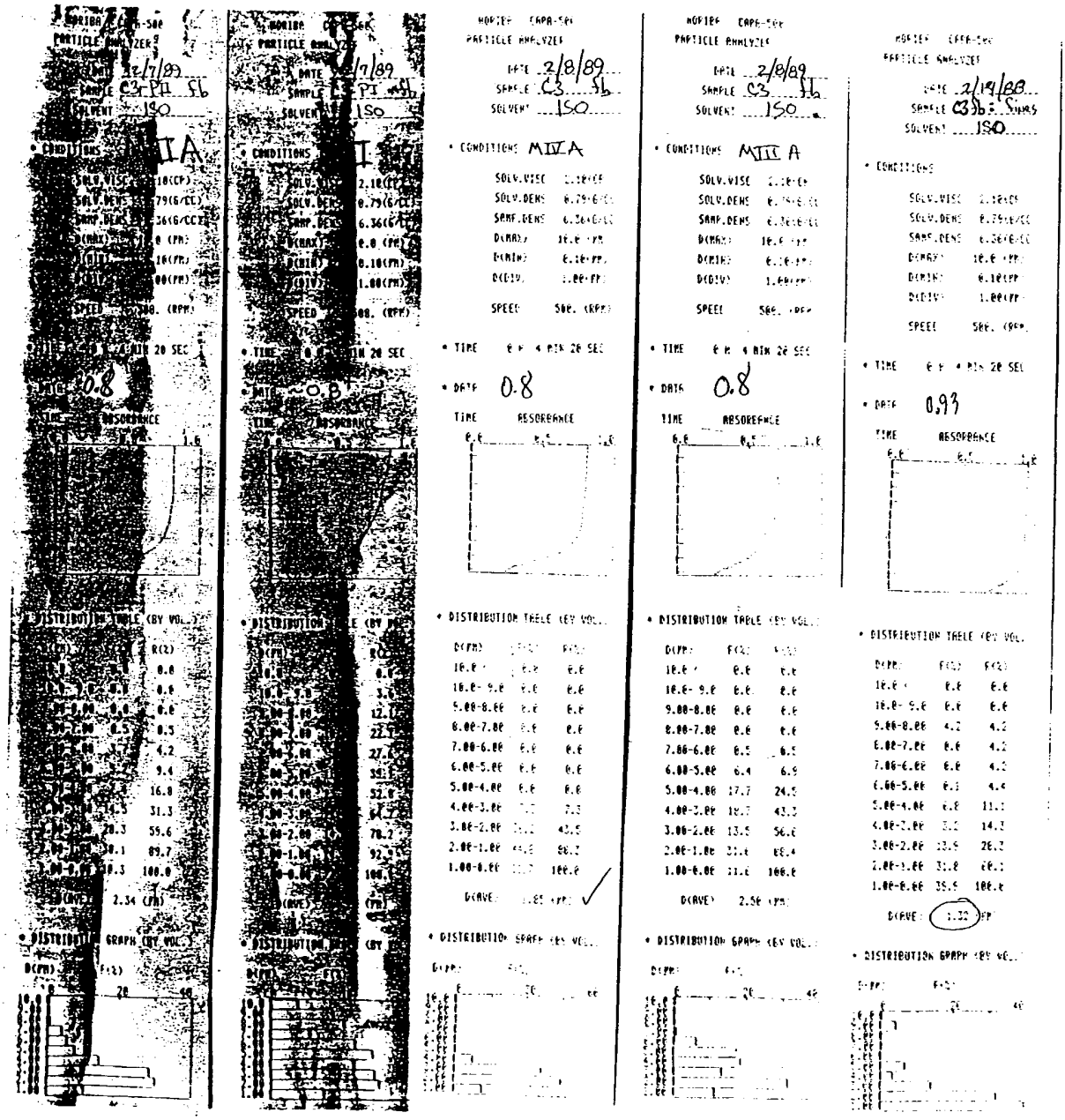
Experiments to look at
Carbonate in 123
Clean O₂ gas with Ascarite
Sample 1 - dense closed
porosity, P ≥ 91%
cut sections from center

Sample 2 open porosity
P < 87%
center section
thin slices aggregate
(2) 3 pellets of each (Peter)
center sections of each
(1) Tem / FELSS (P. Batson)
(2) Magnatometer (T. Moguine)
(3) Induction (Diane Dimos)
(4) CO_2 evolution on dissolution
(5) X ray lattice
(6) XPS



The above understood and witnessed by — Date

and by — Date



HORIBA CAPA-500
PARTICLE ANALYZER
DATE 2/8/89
SAMPLE C3 Sb
SOLVENT ISO
• CONDITIONS MIIA
SOLV.VISC 2.18(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 16.0 (PH)
D(MIN) 0.10(PH)
D(DIV) 1.00(PH)
SPEED 500. (RPM)
• TIME 0 H 4 MIN 20 SEC
• DATA 0.8
TIME ABSORBANCE
• DISTRIBUTION TABLE (BY VOL.)
D(PH) F(%) R(%)
16.0 - 9.0 0.0 0.0
9.00-8.00 0.0 0.0
8.00-7.00 0.0 0.0
7.00-6.00 0.0 0.0
6.00-5.00 0.0 0.0
5.00-4.00 0.0 0.0
4.00-3.00 7.3 7.3
3.00-2.00 35.2 42.5
2.00-1.00 45.8 88.2
1.00-0.00 11.8 100.0
D(AVE) 1.85 (PH) ✓
• DISTRIBUTION GRAPH (BY VOL.)

HORIBA CAPA-500
PARTICLE ANALYZER
DATE 2/8/89
SAMPLE C3 Sb
SOLVENT ISO
• CONDITIONS MIIA
SOLV.VISC 2.18(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 16.0 (PH)
D(MIN) 0.10(PH)
D(DIV) 1.00(PH)
SPEED 500. (RPM)
• TIME 0 H 4 MIN 20 SEC
• DATA 0.8
TIME ABSORBANCE
• DISTRIBUTION TABLE (BY VOL.)
D(PH) F(%) R(%)
16.0 - 9.0 0.0 0.0
9.00-8.00 0.0 0.0
8.00-7.00 0.0 0.0
7.00-6.00 6.5 6.5
6.00-5.00 6.4 6.9
5.00-4.00 17.7 24.5
4.00-3.00 18.7 43.3
3.00-2.00 13.5 56.8
2.00-1.00 21.6 88.4
1.00-0.00 11.6 100.0
D(AVE) 2.56 (PH)
• DISTRIBUTION GRAPH (BY VOL.)

HORIBA CAPA-500
PARTICLE ANALYZER
DATE 2/19/89
SAMPLE C3Sb: Sonic
SOLVENT ISO
• CONDITIONS
SOLV.VISC 2.18(CP)
SOLV.DENS 0.79(G/CC)
SAMP.DENS 6.36(G/CC)
D(MAX) 16.0 (PH)
D(MIN) 0.10(PH)
D(DIV) 1.00(PH)
SPEED 500. (RPM)
• TIME 0 H 4 MIN 20 SEC
• DATA 0.93
TIME ABSORBANCE
• DISTRIBUTION TABLE (BY VOL.)
D(PH) F(%) R(%)
16.0 - 9.0 0.0 0.0
9.00-8.00 4.2 4.2
8.00-7.00 0.0 4.2
7.00-6.00 0.0 4.2
6.00-5.00 0.1 4.4
5.00-4.00 6.8 11.1
4.00-3.00 3.2 14.3
3.00-2.00 12.0 26.3
2.00-1.00 31.8 58.1
1.00-0.00 35.9 100.0
D(AVE) 1.22 (PH)
• DISTRIBUTION GRAPH (BY VOL.)

84

94-95
Sonic

95

The above understood and witnessed by

Date

and

Date

2/7/89

ρ < 87% PI mill $\overline{D}$ = 4.21 μm (quick)
3333/28500
Pre wght: 3.46 (of 3.5) some ~~bounding~~ deformation during isopressing, but Δ wght minimal so ρ density can be deduced.
Post 975C 1h
3.41 1.402 0.448 0.69 4.94 (77.7) → 78% 4.2 too low

PII mill $\overline{D}$ = 2.34 μm (slow)
Pre 3900/29,000
3.46 1.453 0.543 0.9 3.84 60.5 ρ reasonable
3.40 1.285 0.456 0.59 5.76 90.6 <NEED SLIGHTLY HIGHER>

Tomorrow → will mill ρN

~~Pre~~

Yields: ~~Pre~~ 50 MI : Post 48g gap B

gap A { 24 MIIA: 20g 20.5
16 MIIIA: ~~Pre~~

error in pre-weights

78 M4A | D = 1.85 PRE - 4000/30,000 [20°/mm to 675, 10°/mm to 975, 1 h 975C ? quench]

P3	(3.40)	1.452	.543	0.9	3.78	59.4	
	~~3.38~~						
	3.48	1.265	0.460	0.578	6.02	94.6	vent
P4	(3.41)	1.462	.540	0.91	3.75	59.—	
	3.60	1.274	0.468	0.596	6.04	95—	hoing
P5	(3.93)+	1.457	0.561	0.955	3.67	57.7+ → ≥ 58	
	3.60	1.280	0.492	0.62	5.81	91.4+	
	3.65				5.89	92.6	{Probably even higher

MIFB - 4000/30,000 30 μm

P6	(3.55)	1.463	0.508	0.85	4.17	65.6	
	3.53	1.36	0.464	0.674	5.24	(82.4)	vent.
P7	(3.54)	1.462	0.504	0.85	4.165)	65.5	
	3.59	1.358	0.462	0668	5.36	(84.5)	hoing
P8	(3.56)	1.463	0.507	0.85	4.19	65.9	
	3.73	1.352	0.453	0.65	5.73	90.2	

* 1st laminates on 1 side, not severe (must be 'repelletized' pellet)

The above understood and witnessed by: Date and Date

6:15

P9 4000/30000

3.57	1.462	0.517	0.87	4.1	64.5
3.56	1.354	0.463	0.666	5.35	84.-

P10 fines 4/3 as above

1.53	1.436	0.254	0.411	3.72	58.5	was expected
1.54	1.254	0.207	0.256	6.02	94.7	desired back grad

Pellet cutting NEXT (see pg 80 for flow overview)

Pellet 3 & 4 dedicated to vertical & horiz. slicing

Pellets 6 & 7

From tangent saw cut edge: 1 mm slices are 0.055" w/ blade 1.2 .06

Low Density Vertical slices: 1 1.2 slices } 7 altogether + end piece
6 1.0 slices } (marker)
end polished end

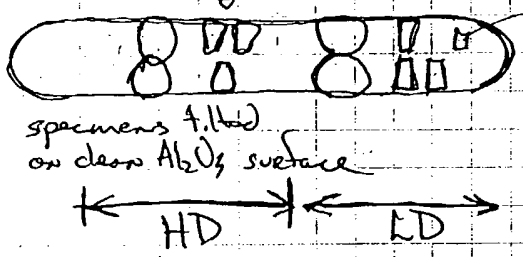
Horizontal: 2 1.0 slices mid-section
1 0.5 top
1 1.0 bottom

High Density
VERTICAL: 5 slices (1 end lost to chipping) 1 end polished
~2mm 1 polished thick chunk
~1.2mm 3 oxygenated
1 saved
HORIZ: 2 mid section
1 ea top & bottom (top chipped)

The above understood and witnessed by _____ Date _____ and by _____ Date _____

8/9/89

OXYGENATION DIAGRAM & SCHEME



2/14 IN AND to 600C @ 20C/MIN; 10C/MIN to 800C

15 MIN SOAK AND start RAMP to 600C @ 0.17°/MIN (10°/h)

To 600C @ 1:30 p.m. 2/15/89 IN dry, CO_2-free O_2.

Pellet (1) O&C

1 mm vertical slices  1°/mn to 850C, 10°/h to 600 2 day (48h), quench.

1) ✓ 1 slice for Tracy Brews (Ramrow)
2) ✓ 1 slice for Alex for XPS of fracture surface
3) save remainder for future use (dessicated)
2A) 1 xtra slice oxygenated

Pellet (2) O&C

1 mm horizontal slices 4 w/ outer slices discarded

1) 1 slice (1.0 mm) ground to .45 mm & cut 3 3mm discs w/ ultrasonic in isopropyl Alc (if possible).
   1 disc to Tom
   2 discs for TEM
   1 spare

2) 1 slice dedicated to Y vs T to Dave's specs

Pellet (3) O&C

spare for (1) x-ray lattice
(2) CO_2 evolution

Administrative Notes

2/16/89

C4 Synthesis Preparations/Notes (ref book IV, pg 46 & pg 14)

	oxide wt. frac.	atomic % Y, Ba, Cu	oxide M.W
Y_2O_3:	0.17(51)	.17	225.81
BaO:	0.46(59)	.33	153.34
CuO:	0.36(25)	.5	79.54

Go with the new!

Example Calc: wt frac Ba Y Cu

Y_2O_3 $225.81 \times \frac{.17 \text{ m}}{\text{mole}} = \frac{38.39}{2} = 19.19$ $19.19/109.56 =$ oxide wt frac $0.17\,51$

BaO $153.34 \times .33 = 50.60$ $50.6/\ \ = 0.4618(5)$

CuO $79.54 \times .5 = 39.77$ $39.77/\ \ = 0.363$

109.56 0.9999

17.51 g Y_2O_3 1.287 009

46.18 g BaO $\left\{\frac{197.35}{153.34} \times 46.18\right\}$ 59.43 g $BaCO_3$

Y_2O_3 $19.19/.99 = 19.209 \rightarrow 19.21$ $\Rightarrow \times 1.5$ $28.81(5)$ 28.82

$BaCO_3$ $50.60(2) \left(197.35/153.34\right) = 65.12(5)$ 97.69 97.69

CuO $39.77/.99 = 39.87$ $59.71(5)$ 59.72

$97.69(0.777) = 75.91 - 97.69 =$

186.23

-21.78 CO_2

164.45

AR

Administrative Notes

FINAL Batch Size for reasonable bulk handling

Y_2O_3 17.51/.999 = 17.52(7) ≈ 17.53

$BaCO_3$ 65.12(5)/.9999 = 65.12(5) ≈ 65.13 ~~343.78~~

Cu_2O 39.77/.999 = 39.80(9) ≈ 39.81

122.47

343.78(7) $BaCO_3$ Y_2O_3 17.53 weighed/transferred

tare 278.65

65.13 ✓ (Δ 0.01?)

~~382.38~~ Cu_2O Mixing yield 122.29 g / 122.47 = 99.85%

t 343.78(7) -Δ 0.15

~~39.78~~ (Δ 0.03)

151.42 OK

tare 1 ~~37.89~~ 73.93

Δ

~~140.48~~ → X bad boat dimensions

tare 2 82.12

58.36 = 122.29 ✓

146.97(6)

88.61

58.36

sintered much more/some liq form

151.42

tare 1 144.37

7.05

87.55

less sintered, less liq

146.97

tare 2 141.74

5.23

88.64

- 12.28 (Cu_2 lost vs 14.53 expected (85% reacted) 2.25 to go)

after GRI 108.71/110. = 98.8% -Δ 1.2%

197.38 (.2)

88.64

108.69

	YBa_2O_3	$BaCO_3$	CuO	SLS / Result
C4-1	0.17	0.33	0.5	assumed stoic pwdr
	0.16	0.36	0.5	analytical determination
C4-2	0.17	0.33	0.5	analytical 1/a
Δ	+0.01	−0.03	—	
C4-3	0.16	0.35	0.5	analytical
Δ	−0.01	+0.02	—	
Δnet	—	−0.01	—	
-3E	0.165	0.35	0.5	
Δ	−0.005	+0.02	—	
Δnet	+0.005	−0.01	—	
C4-4	0.16	0.34	0.5	analytical
Δ	—	−0.01	—	
Δnet	—	−0.02	—	
C4-5	0.16	0.34	0.49	analytical
Δ	—	—	−0.01	
Δnet	—	−0.02	−0.01	Species Implication → trace contam.
C4-6	0.16	0.34	0.478	$C_xO \rightarrow Cu_2O$
Δ	—	—	−0.002	$BaCO_3 \rightarrow Ba(OH)_2$
Δnet	—	−0.02	−0.03	$Y_2O_3 \rightarrow Y_2O_3$ X
C4-7	0.16	0.34	0.478	
Δ	—	—	+0.008	
Δnet	—	−0.02	−0.022	↑

C4-7 TRANSFORM TO STOIC BASIS

Y_2O_3	$BaCO_3$	CuO	
0.17	0.31	~~0.478~~ 0.48	Correct for final batch calc

∴ Y_2O_3 is good as received ✓

$BaCO_3$ is Barium rich by 0.02 at%

CuO is Copper rich by 0.02 at%

A3

Administrative Notes

Post Cal II ~60hs @ 950C in O_2 3/27

195.52
88.64 → post 88.64 (0.2g RXN.)

106.88 − 108.69 = 1.81/2.25 = 80.5% of remainder

12.28
14.09/14.53 theor. = 97%+ loss

Minimal liq. formation.

99.57g yield (due to contamination)

{further contamination upon re-submission to tube for cal III reduces yield further

Peaks @ ~30.2, 29.4, 28.5 2θ Guinier. May be $BaCl_2$, but could also be 2 Φs

$n\lambda = 2d \sin\theta \Rightarrow d = \frac{\lambda}{2}\sin\theta$ 100 = 3.72 something wrong here

3.72 = .775 (sin θ)

4.803 = $\sin^2\theta$

4/19

From Flechaty: $Y_{0.96}Ba_{2.16}Cu_3(Y_{0.32}Ba_{0.72}Cu_1)O_{2.2}$

In reference to VARIATIONAL study: $Ca_{1.16}^{\pm 0.01}\ 0.36\ 0.5 = 1.02$ ± 0.005

A9

Administrative Notes

AB

Administrative Notes

2/17/89 page 74 [redacted] 975C "Property Pellets for [redacted] variation study. - O_2

2g pellets should give ~~ess~~ of material for 4.4 mm thick sintered body - allowing a slice to be cut w/ an interior & exterior surface.

7.5 g #1 stock 1.92 used for pellet (.17 .33 .5)

7.6 g #2 stock 1.90 ↓ ↓ (.15 .33 .52)

8.5 g #3 stock (.17 .35 .48)

9 #4

7.7 #5 to 600C @20°/min; 10°/mw to 975C 4:31-625C
3600/29,000 Quench 1h ~5:10

#1 P3						
	1.92	1.1440	0.479	0.489	3.93	61.8
	1.90	1.0	0.4	0.314	6.05	95—
#2 P3						
	1.91	1.126	0.490	0.488	3.91	61.5
	1.88(?)	0.98	0.412	0.311	6.045	95—
#3 P2		1.133	0.503	0.507	3.85	60.5
	1.95	0.998	0.43	0.536	5.51	86.6
-0.1	1.85					
#4 P2		1.139	0.481	0.490	3.88	61—
	1.90	1.05	0.43	0.37	5.08	79—
	1.86					
#5 P3		1.145	0.46	0.474	3.99	62.7
	1.89	0.993	0.388	0.300	6.2	97.5
	1.86	~~+.05~~	~~0.4~~			

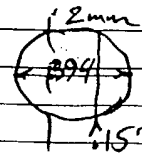
Administrative Notes 2/27

#1-5

P2&3 slicing & oxygenation @ 600C for 66hrs in O2

0.394 inches 0.157 in for center cut

.08 ~ 2 mm



#1 P4 Pre 63.5%

Post 1.85 1.025 0.406 0.335 5.52 87

2/28 Property Pellet Summary (to date)

		stoic	liq	X	X	CaO	
	t	#1	#2	#3	#4	#5	
oxygenated	975	(95)	95	(87)	(79)	97.5	R3
no furnace 2/28	950 II'	(84)	(94)	—	—	(87)	R2
	950	87	R done				R4
MICRO STRUCTURES	ORIG 950	91	'97'	71	78	92	Dawes

950 II #1 & #5 pellets to temp @ 600C after 10°C/min Ramp @ 6:15 pm

> oxygenation run

Out 10:00 A.M. 3/2 40 h O2

$5cc \times \frac{6.36g}{cc} \times = 31.8g$

$\frac{\pi(2.54)^2}{4} X = 5cc$

$5.07 cm^2 X = 5cc$

$X = 5cm^3 / 5.07 cm^2$

$X \cong 1$ cm or 1/2 inch - 1 inch with shrinkage

Administrative Notes

4/24/89

#1 - C's wt% Cu 28.7

at% holes 36.0

Hole Concentration Conversion formula:

Data: wt% Cu (total): 28.7

wt% holes : 36.-

$$\frac{\text{at\% holes} - \text{at\% Cu}_{\text{TOT}}}{\text{Cu}_{\text{TOT}}} = \frac{36-28.7}{28.7} = 0.254$$

average over Valence

see page after next

∴ add Cu valence (2) = 2.25 = average valence Cu

$2.25\,(\text{Cu}_{\text{TOTAL}}) = 2.25\,(3) = 6.75$ total Cu val

+ 7.00 total Ba + Y val (2·2 + 3 = 7)

13.75

↓

total charges

from Squash: Y. $\text{Ba}_2\text{Cu}_3^{2.25}\text{O}_x$

Take total charges & divide by two for O^{2-}

13.75/2 = 6.88 O atoms ⇒ $\text{Y Ba}_2\text{Cu}_3^{2.25}\text{O}_{6.88}$

Administrative Notes

Notes to Kristy concerning Pellet Forming precalculations

To estimate pellet/powder weight for pellet pressing:

A. take die dia & approx. height desired

1. calculate volume in cc. $\left(1.2 \frac{(1.22\text{cm})^2}{4} \times 0.35\text{cm} \times \pi\right) = 0.41\text{cc}$

B. Assume some reasonable 'green' density (unfired pressed pellet) 0.6-0.8 (60-80%) usual for metals > 0.70 w/ small ave. part. dias. (i.e. 3 max).

$(0.41\text{cc} / 0.8) \times 9.0 \frac{\text{g}}{\text{cc}} \approx 3\text{g}$ of powder. (cc density theoretical)

I pressed @ between 16,000 & 20,000 psi.

low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

$X \approx 4{,}000$ for 0.48" dia. die.

Administrative Notes

K.V.F

K. Kroll

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/14/89
SAMPLE α-Al_2O_3
SOLVENT ISO

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	3.97(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 7 MIN 36 SEC

• DATA D~ 0.9

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

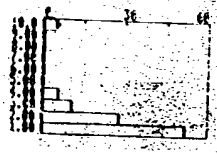
D(PH)	F(%)	R(%)
10.0 <	0.0	0.0
10.0- 9.0	4.5	4.5
9.00-8.00	0.0	4.5
8.00-7.00	0.0	4.5
7.00-6.00	0.0	4.5
6.00-5.00	0.0	4.5
5.00-4.00	0.0	4.5
4.00-3.00	5.5	10.0
3.00-2.00	18.5	28.6
2.00-1.00	20.0	48.5
1.00-0.00	51.5	100.0

D(AVE) 6.57 (PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)

0 30 60



HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/14/89
SAMPLE α-Al_2O_3
SOLVENT ISO

• CONDITIONS

SOLV.VISC	2.10(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	3.97(G/CC)
D(MAX)	10.0 (PH)
D(MIN)	0.10(PH)
D(DIV)	1.00(PH)
SPEED	500. (RPM)

• TIME 0 H 13 MIN 17 SEC

• DATA D~ 0.95

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

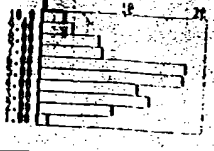
D(PH)	F(%)	R(%)
10.0 <	8.5	8.5
10.0- 9.0	2.8	11.3
9.00-8.00	3.8	15.1
8.00-7.00	7.0	22.1
7.00-6.00	7.4	29.6
6.00-5.00	17.6	47.2
5.00-4.00	17.3	64.5
4.00-3.00	11.9	76.4
3.00-2.00	13.4	89.8
2.00-1.00	9.0	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (PH)

• DISTRIBUTION GRAPH (BY VOL.)

D(PH) F(%)

0 30 60



5/1/89

Administrative Notes

Analytical Results for C3 HD/LD STUDY - Holes

IBM RESEARCH CENTER | ANALYTICAL LABORATORY

IBM

Request for Analysis

Use Ball Point Pen

REQUESTOR T. Shaw PROJECT NO REQUEST NO

DEPARTMENT LOCATION ROOM 25-225 PHONE

REQUESTOR'S SAMPLE IDENTIFICATION HDox, LDox

APPROXIMATE COMPOSITION AND HISTORY OF SAMPLE YBa Cu Oxide

ANALYSES REQUESTED

ANALYSIS METHOD

Cu

ANALYTICAL RESULTS

	HD	LD
wt% "Holes"	33.5	34.2
→ wt% Cu	(22.5)	22.6
	2.21	2.26
	6.81	4.689

tot H₂ → %

%

Aster, Reox: HDox 28.7 wt% Cu ← 28.6
pump @ 300C 36 holes

DATE SUBMITTED 3/22/89 DATE REPORTED 3/24/89 NOTEBOOK REFERENCE YBCO Rpt 121

ANALYST T. Shaw APPROVAL

No

Administrative Notes

Notes to Kristy concerning Pellet Forming precalculations

To estimate ~~pellet~~ pack weight for pellet pressing:

A. take die dia & approx. height desired

1. calculate volume in cc. $\left(1.0 \frac{(1.22\text{cm})^2}{4} \times 0.35\text{cm} \times \pi\right) = 0.41\text{cc}$

B. Assume some reasonable 'green' density (unfired pressed pellet) 0.6-0.8 (60-80%) usual for metals > 0.70 w/ small ave. part. dias. (i.e. 3 max).

$(0.41\text{cc} / 0.8) \times 9.0 \frac{\text{g}}{\text{cc}} \approx 3\text{g}$ of powder.

(9.0 ← density theoretical)

I pressed @ between 16,000 & 20,000 psi.

low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

$X \approx 4{,}000$ for 0.48" dia. die.

Administrative Notes

4/24/89

#1 - C3 : wt% Cu 28.7

wt% holes 36.0

Hole Concentration Conversion Formula:

Data: wt% Cu (total): 28.7

wt% holes : 36.-

$$\frac{\text{wt\% holes} - \text{wt\% Cu}_{tot}}{\text{Cu}_{tot}} = \frac{36-28.7}{28.7} = 0.254$$

average 'over' valence

see page after next

∴ add Cu valence (2) = 2.25 = average valence Cu

$2.25\,(\text{Cu}_{TOTAL}) = 2.25\,(3) = 6.75$ total Cu val

$+ 7.00$ total Ba + Y val (+2·2, +3, 4)

13.75 ⑦

↑ from formula 2.25

$Y_1 Ba_2 Cu_3$

↓ total charges

Take total charges & divide by two for O^{2-}

$13.75/2 = 6.88$ O atoms $\Rightarrow Y Ba_2 Cu_3^{2.25} O_{6.88}$

Administrative Notes

Notes to Kristy concerning Pellet Forming precalculations

To estimate pellet powder weight for pellet pressing:

A. take die dia. & approx. height desired

1. calculate volume in cc. $\left(1.2 \frac{(1.22\text{cm})^2 \times 0.35\text{cm} \times \pi}{4}\right) = 0.41\text{cc}$

B. Assume some reasonable 'green' density (unfired pressed pellet) 0.6-0.8 (60-80%) usual. for metals > 0.70 w/ small ave. part. dias. (i.e. 3 mm).

$(0.41\text{cc} / 0.8) \times 9.0 \frac{\text{g}}{\text{cc}} \approx 3\text{g}$ of powder.

(9.0 g/cc = density theoretical)

I pressed @ between 16,000 & 20,000 psi.

low side for pure metal ∴

$\frac{X}{(\text{dia die})^2}$ = desired pressure where X = 1" scale pressure

$X \approx 4{,}000$ for 0.48" dia. die.

Administrative Notes

K. Kroll

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/9/89
SAMPLE ASSC-6
SOLVENT ISO

• CONDITIONS

SOLV.VISC	2.18(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	2.61(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 13 MIN 17 SEC

• DATA 0.95

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	8.5	8.5
10.0- 9.0	2.8	11.3
9.00-8.00	3.8	15.1
8.00-7.00	7.2	22.3
7.00-6.00	7.4	29.6
6.00-5.00	17.6	47.2
5.00-4.00	17.3	64.5
4.00-3.00	11.9	76.4
3.00-2.00	13.4	89.8
2.00-1.00	9.0	98.8
1.00-0.00	1.2	100.0

D(AVE) 4.84 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

10 20

HORIBA CAPA-500
PARTICLE ANALYZER

DATE 3/14/89
SAMPLE α-Al2O3
SOLVENT ISO

• CONDITIONS

SOLV.VISC	2.16(CP)
SOLV.DENS	0.79(G/CC)
SAMP.DENS	3.97(G/CC)
D(MAX)	10.0 (PM)
D(MIN)	0.10(PM)
D(DIV)	1.00(PM)
SPEED	500. (RPM)

• TIME 0 H 7 MIN 36 SEC

• DATA D~ 0.9

TIME ABSORBANCE

0.0 0.5 1.0

• DISTRIBUTION TABLE (BY VOL.)

D(PM)	F(%)	R(%)
10.0 <	0.0	6.6
10.0- 9.0	4.5	4.5
9.00-8.00	0.0	4.5
8.00-7.00	6.6	4.5
7.00-6.00	0.0	4.5
6.00-5.00	0.0	4.5
5.00-4.00	0.0	4.5
4.00-3.00	5.5	10.0
3.00-2.00	10.5	20.6
2.00-1.00	25.8	46.5
1.00-0.00	51.5	100.0

D(AVE) 6.52 (PM)

• DISTRIBUTION GRAPH (BY VOL.)

D(PM) F(%)

10 20

5/1/89

Administrative Notes

Analytical Results for C3 HD/LD STUDY - Holes

IBM RESEARCH CENTER | ANALYTICAL LABORATORY

IBM

Request for Analysis

Use Ball Point Pen

REQUESTOR T. S Fisher PROJECT NO REQUEST NO

DEPARTMENT LOCATION ROOM 25-223 PHONE

REQUESTOR'S SAMPLE IDENTIFICATION HD ox , LDox

APPROXIMATE COMPOSITION AND HISTORY OF SAMPLE Y Ba Cu Oxide

ANALYSES REQUESTED

ANALYSIS METHOD

low

ANALYTICAL RESULTS

	HD	LD
wt% "Holes"	33.5	34.2
wt% Cu	(27.5)	27.0
	2.31	2.36
	6.81	4.689

~~Tot H — %~~

~~ppm~~

Aster, Reox : HDox 28.7 wt% C ← 286
pure O2 300C 36 holes

DATE SUBMITTED 3/22/89 DATE REPORTED 3/24/89 NOTEBOOK REFERENCE 1/16 24/121

ANALYST JC Chen APPROVAL

No

5/21/07

Administrative Notes

Recalculation Pre HD, LD values w/ 28.7% Cu

HD holes 33.5 ∴ $\frac{33.5-28.7}{28.7} = 0.167$

$2 + 0.167 = 2.167\ (3) = {}^{+}6.50$

$+\ 7.$

$13.50 / 2 = 6.75 \Rightarrow \therefore Y Ba_2 Cu_3^{2.167} O_{6.75}$

LD $\frac{34.2-28.7}{28.7} = 0.192$ $\quad 2.192\ (3) = 6.58$

$+7$

$13.58 / 2 = 6.79$

$\therefore Y Ba_2 Cu_3^{2.192} O_{6.79}$

with orignal anal. Cu values

HD $\frac{33.5-27.5}{27.5} = 0.22$ $\quad 2.22\ (3) = 6.66 + 7 = 13.66 / 2 = 6.83$

LD $\frac{34.2-27.0}{27} = 0.27$ $\quad 2.27\ (3) = 6.81 + 7 = 13.81 / 2 = 6.90(5)$

w/ ave. $27.5 + 27 = 27.25$

LD $\frac{34.2-27.25}{27.25} = 0.25(5)$ $\quad 2.255\ (3) = 6.765 = 13.765 / 2 = 6.88$

HD $\frac{33.5-27.25}{27.25} = 0.23$ $\quad 2.23\ (3) = 6.69$ ~~6.845~~ Ca.

Composition #	Rel Pellet* density (%)	Actual density	Green Rel δ	Green act δ
1	84 (90.8)	5.34	62.9	4.0
2	98.9	5.95	61.5	3.90
3	71	4.52	60	3.82
4	77.8	4.95	62	3.93
5	87 (91)	5.54	63.4	4.03

* after 1h sinter @ 950C